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APPLICATION NUMBER: 60/533,824

FILING DATE: *December 31, 2003*

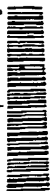
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Approved for use through 10/31/2002. OMB 0651-0032

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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

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<input type="checkbox"/> Additional inventors are being named on the _____ separately numbered sheets attached hereto.				
TITLE OF THE INVENTION (280 characters max)				
TEST APPARATUS FOR DIRECT MEASUREMENT OF EXPANSION AND SHRINKAGE OF OIL WELL CEMENTS				
Direct all correspondence to: CORRESPONDENCE ADDRESS				
<input type="checkbox"/> Customer Number		<div>Place Customer Number Bar Code Label here</div>		
OR Type Customer Number here				
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ENCLOSED APPLICATION PARTS (check all that apply)				
<input checked="" type="checkbox"/> Specification Number of Pages		96	<input type="checkbox"/> CD(s), Number	
<input type="checkbox"/> Drawing(s) Number of sheets			<input type="checkbox"/> Other (specify)	
<input type="checkbox"/> Application Data Sheet. See 37 CFR 1.76				
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT (check one)				
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27.				
<input checked="" type="checkbox"/> A check or money order is enclosed to cover the filing fees				
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FILING FEE AMOUNT (\$) \$160.00				
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.				
<input checked="" type="checkbox"/> No.				
<input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are: _____				

Respectfully submitted,

SIGNATURE

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Date: December 31, 2003

REGISTRATION NO.

25,928

(if appropriate)

Docket Number:

206,405

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop Provisional Application, Assistant Commissioner for Patents Alexandria, VA 22313-1450.

031431 U.S. PTO
60/533824

Jennings, S.S.
AFS: 206,405
12/30/03 (jfb)

TEST APPARATUS FOR DIRECT MEASUREMENT OF EXPANSION AND SHRINKAGE OF OIL WELL CEMENTS

Field of the Invention

The invention relates to a laboratory test method and apparatus for determining the amount of shrinkage or expansion of test samples of cement compositions of the type used in cementing oil wells.

Background of the Invention

During oil and gas well drilling operations, it is often necessary to seal the space between the outer casing string, or pipe, and the rock formation through which the drill has passed in order to prevent the undesired flow of fluids including gas, oil and water, in either an upward direction along the well pipe casing or downwardly where it might be dissipated into the formations below and/or mixed with other fluids.

A variety of compositions are pumped into the bore hole in the rock formation for the purpose of providing a tight seal between the metal well casing pipe and the surrounding irregular rock surface. The cement composition is disposed in the form of a highly flowable slurry that will easily pass downwardly through the irregular annulus formed by the casing and surrounding rock. Its particular formulation will depend upon a number of parameters related to the local conditions. Various mechanical devices are available to provide a

mechanical block at the lower-most portion of the bore hole in order to stop the flow of the cement slurry.

The cementing can be required at considerable depths so that the slurry composition is subjected to extremes of high pressure and temperature as it is curing and forming a monolithic mass in the irregular annulus. It is well known in the art that, under these conditions, various cement compositions will either shrink or expand to some extent. Since the cement compositions are often formulated at the site of the well in order to accommodate the specific subterranean conditions present and the downhole rock formations, it is desirable to know in advance how a particular "custom" cement composition will perform with respect to expansion/shrinkage under the subterranean conditions of setting and curing.

It is therefore a principal object of the invention to provide a laboratory test method and apparatus to measure the expansion and/or shrinkage of a cement slurry as it sets and cures under pressure and temperature conditions simulating those in the well in which the particular cementing composition is to be utilized.

It is known in the prior art to provide various static forms for laboratory testing to measure expansion of cement formulations upon curing. Other types of apparatus and forms are known for determining the shrinkage.

It is an object of the present invention to provide a single laboratory test apparatus that is capable of measuring both expansion and shrinkage.

It is another object of the invention to provide a reliable, sturdy test apparatus that can be reused with minimal preparation and cleaning between uses.

Yet another object of the invention is to provide a laboratory test and method apparatus that is capable of measuring both expansion and shrinkage of a cement composition for which there is no previous data as to whether the cement composition is likely to shrink or expand upon curing.

Summary of the Invention

The apparatus of the invention includes a modified ultrasonic cement analyzer cell sleeve. The unmodified cell is commercially available from Chandler Engineering Company of Tulsa, Oklahoma, www.chandlereng.com. A cement slurry is poured into the bottom of the test cell to a predetermined level. A rubber diaphragm is placed above the level of the cement. A water cushion is added to the cell above the rubber diaphragm. The floating piston is attached to a linear displacement transducer (LDT) and is pressure-balanced in the water above the rubber diaphragm.

The purpose of pressure balancing the floating piston attached to the LDT is to avoid adding any additional pressure forces.

The upper portion of the cell cap contains the expansion/shrinkage mechanical measuring devices which record a positive or negative volume change in the hardened cement.

As the setting cement undergoes any volume changes, the movement of the LDT is recorded digitally as a percentage value of shrinkage or expansion of the original volume.

The system advantageously includes a processor/controller that is linked to a programmed general purpose computer that is programmed to receive basic sample identification information, starting and completion times and the final value as a percentage,

along with as many intermediate readings and times as may be desired for analytical purposes. The program is within the ordinary skill of the art and does not itself constitute the invention. The apparatus provides the advantage of a direct, immediate display/recorded value of the expansion or shrinkage of the sample cement composition.

Detailed Description of A Preferred Embodiment

Attached is a description of one preferred embodiment of the apparatus of the invention, the operation of which apparatus also illustrates the method.

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Components Section Includes:

None

Reference Section Includes:

Abbreviation List
Warranty Statement
Comment Card
Sales Literature

General Information

Introduction

Purpose and Use

The 4268ES Cement Expansion/Shrinkage Cell is used as an accessory to a Model 5265 Static Gel Strength Analyzer (SGSA) or Model 4265 Ultrasonic Cement Analyzer (UCA). When combined with a precision pressure controller (Model 6265-I), the system continuously measures the expansion or shrinkage of a cement sample under high temperature and high-pressure conditions.

The system measures the change in volume of the sample using a diaphragm and displacement piston combined with a precision LVDT (Linear Variable Displacement Transducer). The resulting translation of the piston is scaled in units of milliliters (mL) or % expansion. The data is presented graphically using the 5270 Data Acquisition and Control System (DACS).

Description

The 4268ES Cement Expansion/Shrinkage Cell makes use of the programmable temperature controller that is a part of the Model 5265 or 4265 instruments. The system is equipped with a programmable pressure controller for the Model 6265-I Intensifier assembly. Using the controllers, multiple segment ramp and dwell temperature and pressure schedules may be defined for the sample.

Features and Benefits

- Easy to install and use.
- Continuously measures the expansion or shrinkage of API Cement slurries under high pressure (10 kpsig, 69 MPa max.) and temperature (400°F, 204°C max.) conditions.
- Ability to measure +10% expansion (20mL), -16% shrinkage (32mL).
- Single vessel curing to preserve sample conditions and testing integrity. Sample is cured in the vessel, improving sample and test integrity.
- Programmable temperature and pressure control.
- Cement is isolated from the pressurizing media using a flexible diaphragm
- Uses Chandler Model 5270 Data Acquisition and Control System for data retrieval, analysis, and storage.

Specifications

Measurements:	± 20 mL volume change of the cement sample, sample temperature, sample pressure. Piston displacement may reach -32 mL for initial sample compression
Vessel Volume:	200mL
LVDT Measurement Range:	± 0.500 inches (± 12.7 mm)
Operating Environment:	50°F - 110°F (10°C - 43°C) – non-condensing
Maximum Temperature:	400°F (204°C)
Maximum Pressure:	10,000 psig (69.1 MPa)
Input Voltage:	85 – 240 VAC, 50/60 Hz (LVDT electronics and related data acquisition hardware), see SGSA or UCA instrument manual for separate power requirements
Input Power:	50 VA or less

Section 1 – Installation

Unpacking the Instrument

Remove the instrument from the packing crate carefully. The unit comes fully equipped with all the necessary components and any spare parts that were ordered with the unit. Make sure that no parts are lost when discarding the packing materials. Place the instrument on a firm table, close to the water source and required electrical outlets.

After the instrument is removed from the shipping crate, the equipment and spare parts should be checked against the packing list to ensure that all parts have been received and none are damaged.

Note: File an insurance claim with your freight carrier if damage has occurred during shipping. Verify all parts shown on the enclosed packing list have been received. If items are missing, please notify Chandler Engineering immediately.

Utilities Required

- Power: 85-240 VAC, 50/60Hz (LVDT electronics and DAC hardware)
- Power to Instrument: 230 VAC, 50/60 Hz
- Water: Filtered pressurizing water, 5-150 psig (.34-10.4 bar)
- Coolant: Clean water or Ethylene glycol solution
- Air: Filtered, dry compressed air; 75-125 psig (5.2-8.6 bar)
- Drain: Suitable for hot water

Tools/Equipment Required to Operate Instrument

- 5/8" Wrench
- 7/16" Wrench
- Lithium Grease (or equivalent)
- Bench Vise (minimum jaw opening 5" (13mm))

Installing the Instrument

1. Locate the SGSA or UCA instrument near power, air, water, and drain connections.
2. Connect power to the instrument using the power cord supplied with the analyzer. The power plug may need to be changed if the local receptacle is incompatible with the plug supplied with the instrument. A 10A fuse or circuit breaker is recommended. A 1 KVA uninterruptible power supply (UPS) is recommended to protect the data acquisition electronics and test data from brief power failures.
3. Connect the Intensifier connector labeled "Pressure Control" to the connector labeled "Pressure Control" at the back of the SGSA instrument.
4. Connect the power supply to the connector labeled "Power." The Pressure Control System will emit a normal vibrating sound until the air supply is connected.
5. Connect the air, water, and drain lines to the Intensifier as detailed and shown below. All water and air supplies must be filtered.

DB25P

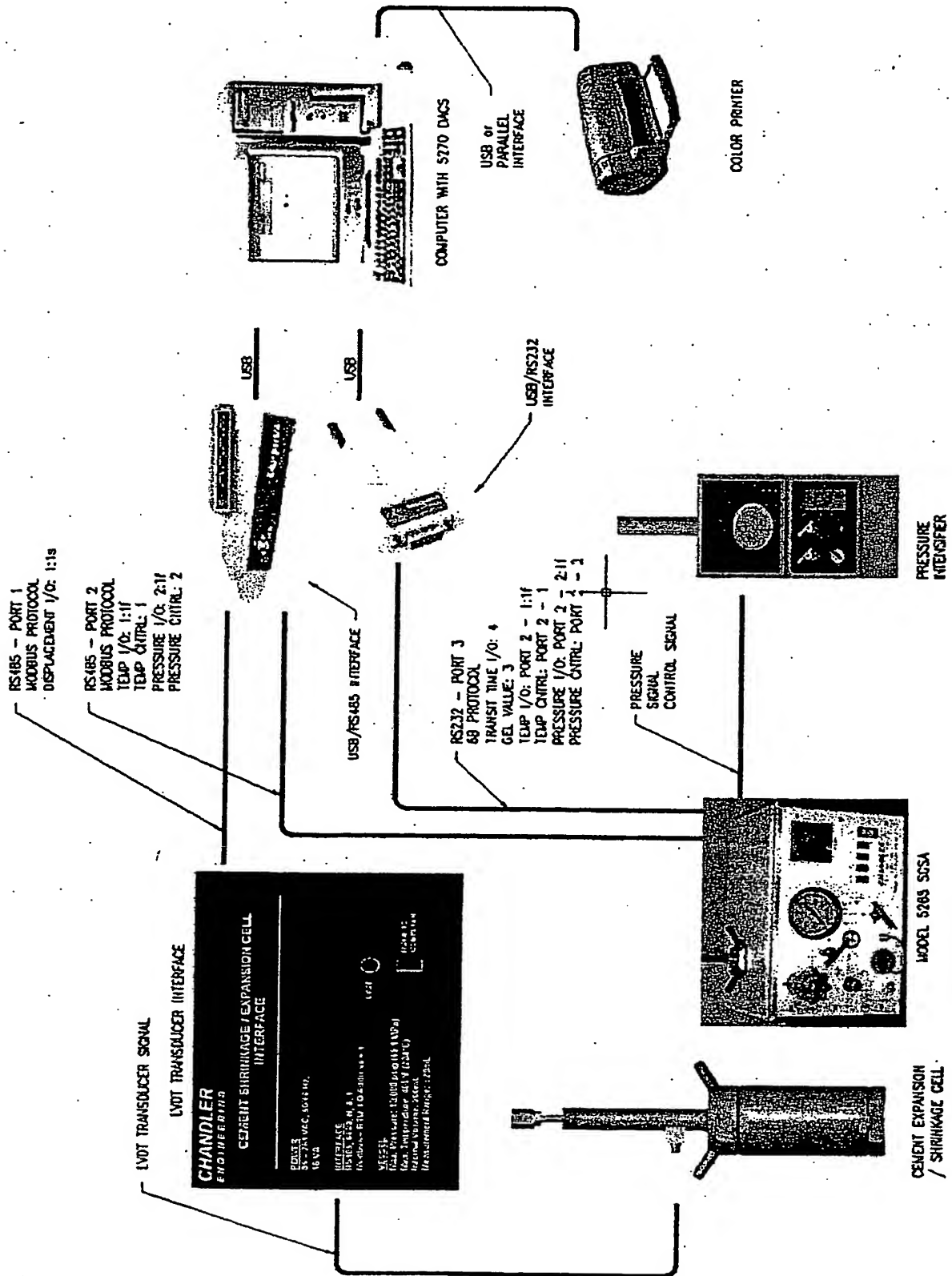
NOTE: *The ports use 1/8" female NPT threaded connections. The NPT ports are adapted for use with 1/4" and 1/8" tubing using Swagelok® fittings. For high pressure connections, the 1/8" stainless steel tubing must have 0.035" wall thickness required for use at 10,000 psig. If metric size tubing is required, the port adapters may be changed to adapt the female NPT ports to metric tube connections. In all cases, the high pressure tubing and connections must be rated for 10,000 psig (69 MPa).*

Connecting the Instrument to the Computer

The system requires three serial interface (two RS485, one RS232) connections to the computer system that is running the 5270 software. These interfaces are supplied using two USB / Serial Port hubs.

- LVDT Interface: RS485 Comm
- Temperature and Pressure Controllers: RS485 Comm
- Model 5265 SGSA: RS232 Comm

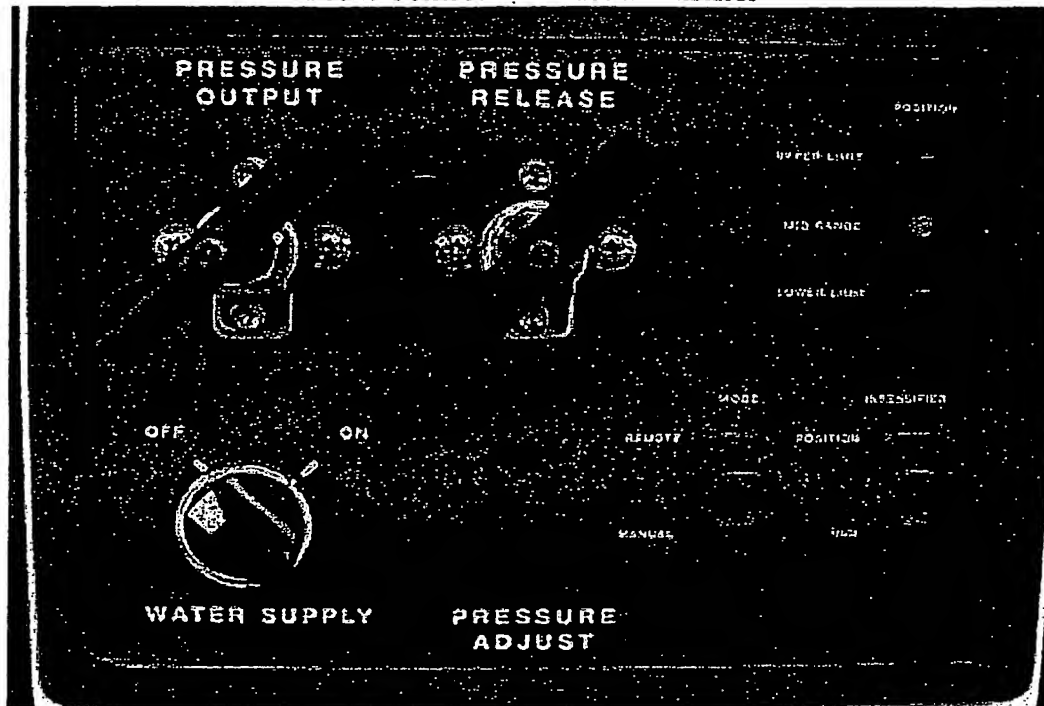
The following diagram summarizes the connections. Please refer to SPR118-1061 and SPR118-1062 in *Section 6 – Drawings and Schematics* for additional details.



Section 2 – Operating Instructions

Description of Valves and Switches

Front Control Panel for Intensifier



For the following descriptions, refer to the Figure above.

Pressure Output Valve

The Pressure Output valve should be opened counterclockwise (CCW) to transmit pressure to the instrument during testing.

Pressure Release Valve

The Pressure Release valve relieves the pressure from the Intensifier unit. If the Pressure Output valve is OPEN, the Pressure Release valve will also relieve the pressure from the instrument. The pressure can be relieved on the Intensifier unit but NOT on the cell by CLOSING the Pressure Output valve BEFORE opening the Pressure Release valve.

Water Supply

The Water Supply switch controls the water source to the Intensifier unit. It should remain in the ON position when the Intensifier unit is operating.

Pressure Adjust Knob

The pressure adjust knob is used to manually increase the pressure in the Intensifier unit when the Mode switch is in the Manual position. Turning the knob clockwise will increase pressure, while a counterclockwise turn will decrease pressure. The pressure value is indicated on the gauges located on the Intensifier and SGSA (or UCA). The pressure value is also recorded by the data acquisition software (5270).

Mode Switch

The mode switch allows the operator to choose between manual and remote mode for control of the Intensifier. Remote control allows the Intensifier unit to be controlled by an external pressure controller.

Intensifier Switch

The Intensifier switch may be placed in either RUN or POSITION. Selection of POSITION allows the piston to be moved to the mid range position in preparation for running a test.

Normally, the Intensifier switch is in the RUN position.

In most cases, a test may be started with the piston at the bottom position. When pressure is applied to the cell the piston will translate slightly as pressure is developed. Once the pressure is static, the piston may be repositioned to the mid position (green light illuminates) by opening the Pressure Release valve slightly. The loss of pressure causes the piston to move upwards. Close the Pressure Release valve when the green light appears.

Position Indicator Lights

The Position Indicator Lights verify the approximate location of the piston in the Intensifier unit (Top, Mid-Range, Bottom). For optimal operation during testing, the piston should be near the mid-range position. If the piston reaches either the minimum or maximum position during testing, it can no longer control the pressure of the system effectively, and must be repositioned.

Note: If the piston position is located between the bottom & mid-range or between mid-range & top, the position indicator light may be OFF.

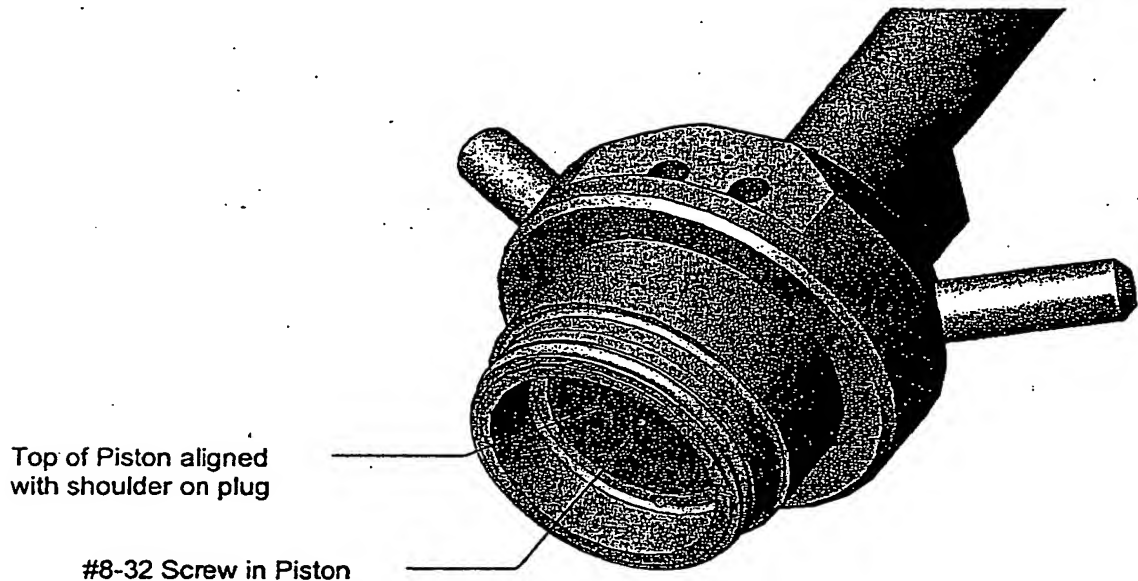
Pressure Vessel Preparation

The pressure vessel consists of a vessel, top plug assembly, bottom plug assembly, diaphragm, translation piston, LVDT transducer assembly, and seals.

To prepare the vessel for use:

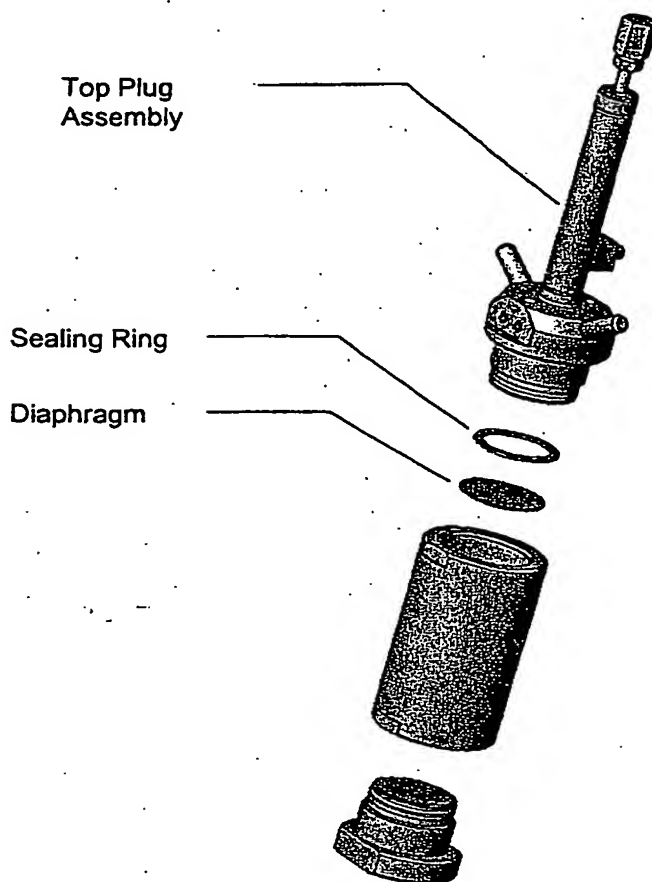
1. Remove the top and bottom plugs from the vessel.
2. Remove and replace the retaining ring, O-ring, and backup ring from each plug if wear is apparent.
3. Clean the vessel and plugs using soap and water solution. Do NOT immerse the Top Plug assembly in water.

4. Position the translation piston until it is aligned with the shoulder on the top plug. Manually press the piston into the plug, use an #8-32 screw in the center of the piston if it must be retracted. Refer to the following illustration.
5. Apply lithium grease to the inner tapered surfaces of the vessel and all surfaces that contact the cement sample.



Filling the Pressure Vessel

1. Prepare the vessel using the "Pressure Vessel Preparation" procedures above.
2. Fill the vessel with slurry up to the shoulder where the diaphragm seals. Do not allow cement to contact the diaphragm sealing surface or the threads.
3. Place a flat diaphragm above the liquid slurry and against the sealing shoulder.
4. Insert the diaphragm sealing ring above the diaphragm.
5. Insert and hand-tighten the top plug assembly. The plug must be tightened to seal the diaphragm against the shoulder.
6. Remove both high pressure port plugs from the top plug.
7. Using a squeeze bottle (or equivalent), fill the volume below the displacement piston (above the diaphragm) until water appears at both high pressure ports.
8. Insert and tighten the thermocouple in the top port.
9. Insert and tighten the remaining top high pressure port with the supplied plug (5/8" wrench).
10. Slide the vessel into the heating and cooling jacket on the instrument.
11. Connect the 1/8" tubing pressure port to the Intensifier. Tighten both ports using a 7/16" open-end wrench.
12. Open the Pressure Output valve on the intensifier. Turn on the water supply.
13. Loosen the high pressure fitting at the top of the LVDT tube to allow trapped air to escape and to allow the LVDT tube to fill with water. Once the tube is full of water, this step may not be required unless the LVDT tube is disassembled or the piston is removed.
14. Connect and hand-tighten the signal cable to the LVDT transducer.



Starting a Test

Once the vessel is filled and installed in the instrument, the temperature and pressure schedules must be configured.

Pressure Control System

The pressure control system is a piston-type intensifier with an approximate ratio of 100:1. It uses air pressure to create hydraulic pressures up to 10,000 psig (69.1 MPa).

The piston in the intensifier must be pre-positioned to approximately mid-range when a test is started to allow bi-directional motion of the piston as the vessel temperature and sample volume vary. Three LED's on the front panel of the pressure control system are used to indicate the approximate piston position.

To start the pressure control

1. Turn ON the water supply switches on the Pressure Control Intensifier.
2. Verify that the manual Pressure Adjustment knob is fully counterclockwise (CCW).
3. Verify that the lower limit red LED is illuminated.
4. Set the Run/Position switch to RUN.

5. Set the Remote/Manual switch to MANUAL.
6. Verify that the Pressure Output valve on the pressure control intensifier is open and the Pressure Release valve is closed.
7. Use the Pressure Adjust potentiometer to increase the pressure. Verify that pressure builds in the vessel and check for leaks.
8. Return the manual Pressure Adjustment knob to the fully counterclockwise (CCW) position, releasing the pressure.
9. Set the Remote/Manual switch to REMOTE.
10. Start the pressure control program in the controller. Wait until 500 psig (approx.) builds in the vessel.
11. Open the Pressure Release valve slightly to allow the piston to translate upwards until the green light on the Intensifier front panel appears. Close the Pressure Release valve.

Ending a Test

To end a test, use the following procedure:

1. The data acquisition software must be stopped and the temperature and pressure controller programs must be stopped.
2. Turn OFF the heater power switch.
3. If the sample temperature is above 212°F (100°C), leave approximately 1000 psig on the sample to prevent water from boiling.
4. Turn ON the cooling water to the vessel.
5. Open the "Pressure Release" valve on the Intensifier.
6. Turn OFF the "Water Supply" valve.
7. Once the vessel has cooled, disconnect the LVDT signal cable and disconnect the 1/8" tube connection on the side of the top plug.
8. Slide the vessel assembly out of the heating jacket.
9. Place the vessel in a bench vise.
10. Remove the top and bottom plugs from the vessel. The same wrench used with a UCA or SGSA vessel may be used.
11. To remove the sample from the vessel, orient the vessel with the label "TOP" upwards. Use a block of wood (or equivalent) with a hammer to drive the sample from the tapered ID vessel.
12. Clean all parts in preparation for the next test.

* To push intensifier to lower limit - switch to (RUN)
 - pressure release cl

MANUAL control - intensifier switch to (RUN) (4)

- remote manual to (MANUAL) (5) /

Section 3 – Maintenance

Tools Required

- 5/8" Wrench
- 1/2" Wrench
- 7/16" Wrench
- Teflon Tape
- Hex wrenches
- Screwdrivers (flat blade, Phillips head)
- Bench Vise

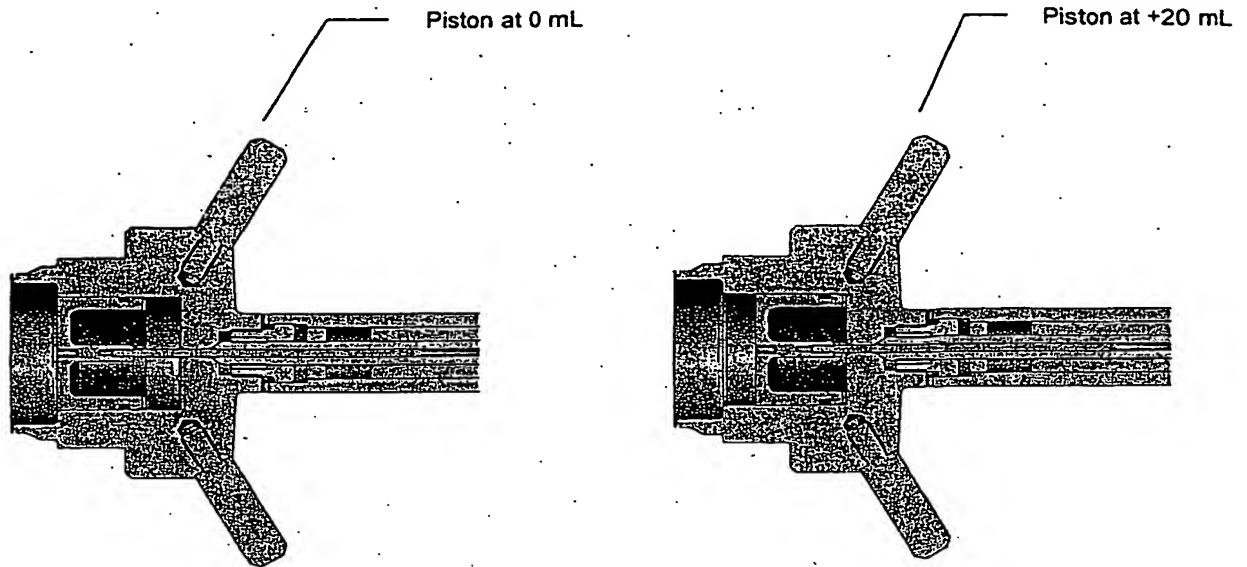
Cleaning and Service Tips

- Keep the test cell surfaces exposed to cement coated with a thin layer of grease, keeping the tips of the transducers free from grease. This reduces the chance of corrosion and prevents cement from adhering to the metal.
- Keep cement off threads and out of the high-pressure ports on the top plug of the test cell.
- Lubricate the threads on the test cell plugs periodically with lithium grease (or equivalent).
- Thoroughly clean test cell of all cement immediately after each test.
- Keep the sensor faces and cavities inside each test cell plug clean and flat.

Calibration Procedure

The temperature and pressure controllers and transducers require periodic calibration. Temperature calibration involves applying known value to the thermocouple electronics using a thermocouple simulator or precision bath. Pressure calibration involves the use of a deadweight tester to provide a known pressure value to the transducers.

The LVDT displacement transducer is connected to the translation piston. When the piston is aligned with the shoulder in the top plug, the output signal is adjusted to 0 vdc. When the piston is fully inserted into the plug (-0.500in), the displaced volume is +20mL. When the piston is extended from the plug shoulder (+0.500in), the displaced volume is -20mL.



Using the Data Acquisition Software, go to the calibration section. For the ZERO setting, position the piston at 0 mL, enter 0 mL as the 5270 calibration value. For SPAN setting, position the piston at +20 mL, enter 20.00 mL as the 5270 calibration value.

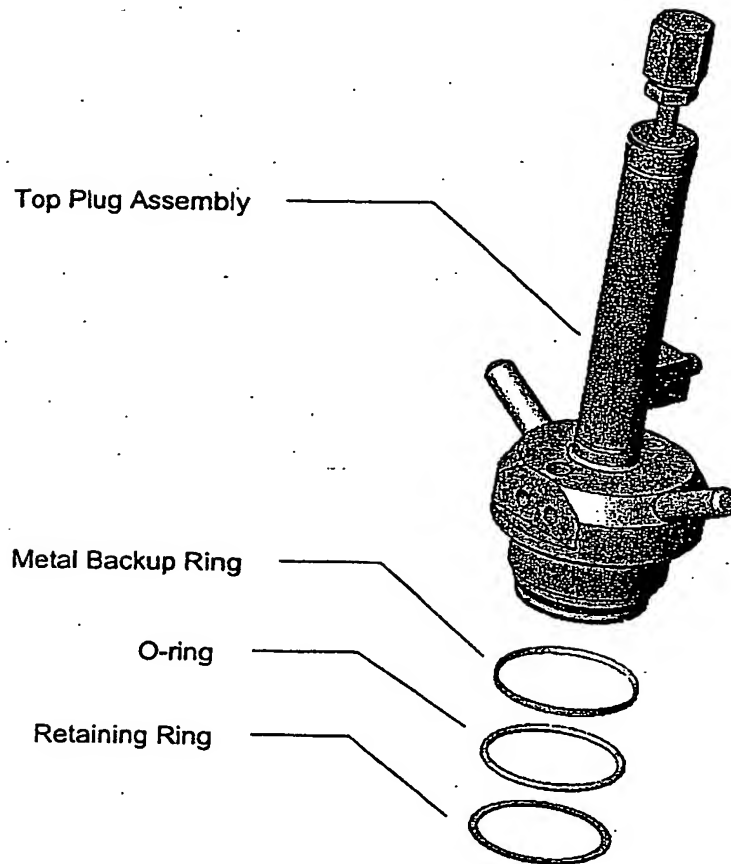
If the system must be calibrated to indicate % displacement the procedure is similar. The total cement sample volume is 200 mL. For the ZERO setting, position the piston at 0 mL, enter 0% as the 5270 calibration value. For SPAN setting, position the piston at +20 mL, enter 10% as the 5270 calibration value. The signal units definition may require a change from mL to % unless both signals were defined when the instrument was configured in 5270.

The specifics related to using the software may be found in the Model 5270 DACS software manual.

Replacing the O-rings

The O-ring and back-up ring should be changed on the top and bottom plugs if there is any evidence of wear.

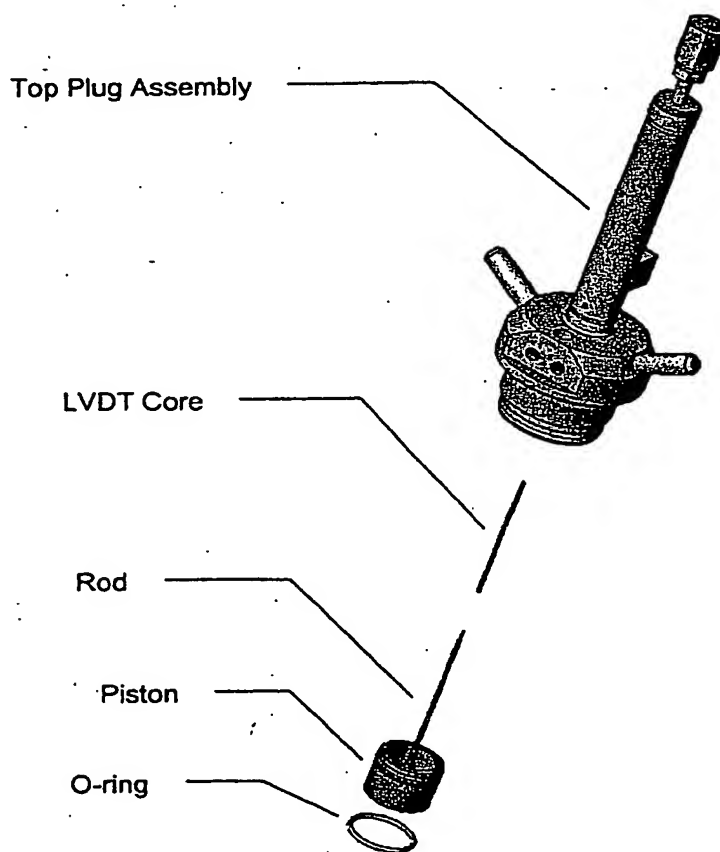
1. Remove the top and bottom plugs from the test cell.
2. Remove the O-ring retaining rings.
3. Clean the metal backup ring. Replace the O-rings. Apply lithium grease (or equivalent) to the O-rings and backup rings. Refer to the following illustration.
4. Remove the displacement piston from the top plug. Replace the O-ring. Lubricate the piston sealing surfaces to allow the piston to move freely. Do not bend the LVDT rod.



Replacing the LVDT Rod and Core

If the LVDT rod or core are bent during disassembly, they must be replaced using the following procedure:

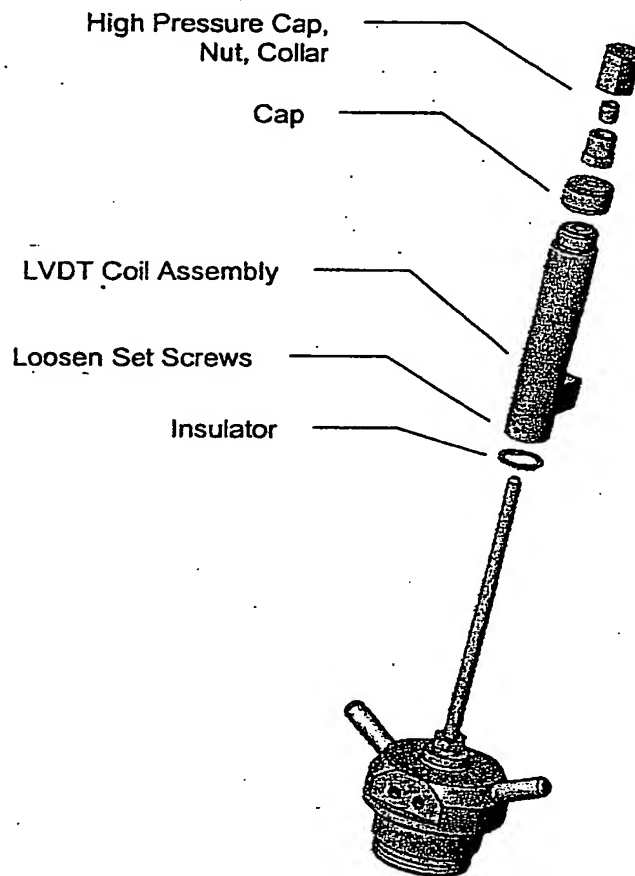
1. Remove the top plug assembly from the vessel.
2. Insert a #8-32 screw into the center hole on the displacement piston.
3. Gently retract the piston and rod assembly.
4. Unscrew the rod from the piston and replace the LVDT rod and Core.



Replacing the LVDT Coil Assembly

If the external LVDT coil assembly is defective, it must be replaced using the following procedure:

1. Disconnect the cable to the LVDT coil assembly.
2. Remove the high pressure cap, nut, and collar from the top of the LVDT tube.
3. Loosen the two set screws at the bottom of the LVDT coil assembly.
4. Gently slide the LVDT coil assembly off of the high pressure tube.



Maintenance Schedule

MAINTENANCE SCHEDULE Model 4268ES Cement Expansion/Shrinkage Cell					
COMPONENT	EACH TEST	MONTHLY	3 MONTHS	6 MONTHS	ANNUAL
Vessel Seals and Diaphragm	Inspect & replace as required				
Vessel Assembly	Clean				
Rupture disc					Replace
Signal Cables	Inspect & replace as required		Inspect & replace as required		Inspect & replace as required
Temperature Signal				● Calibrate	
Pressure Signal				● Calibrate	
LVDT Displacement Signal				● Calibrate	
This maintenance schedule applies to normal usage of two tests per day. Detailed procedures for these operations are contained in your manual. ● Per API Specifications Where Applicable					

Section 4 – Troubleshooting Guide

Problem	Solution
System will not pressurize	<ol style="list-style-type: none"> 1. Verify that the water supply is present and turned ON. 2. Verify that the air supply is present. 3. Verify that the power cable is connected to the intensifier 4. Verify that air is not present in the lines 5. Check for water leaks at the vessel or interconnecting tube fittings. 6. Verify that the piston in the intensifier system is not at the top limit (Red LED is illuminated). 7. LVDT tube or other part of the system is not full of water or trapped air.
Poor sample expansion / shrinkage measurement results	<ol style="list-style-type: none"> 1. Vessel was not filled correctly. 2. Diaphragm is not sealed against the vessel shoulder or is missing the seal ring. 3. LVDT tube is not full of water or has trapped air. 4. Displacement piston position was not aligned with shoulder when top plug assembly was prepared. 5. Volume between diaphragm and piston was not filled with water during top plug preparation. 6. Bent LVDT rod or core. 7. Seal on displacement piston is leaking.
Poor temperature or pressure control	<ol style="list-style-type: none"> 1. Defective thermocouple or pressure transducer 2. Incorrect control program. 3. Incorrect controller configuration parameters. 4. Defective controller or related electronics.

Section 5 - Replacement Parts

Part Number	Description
6265-I	Intensifier System (with internal pressure transducer)
SPR118-1000	Vessel Assembly, Cement Expansion/Shrinkage
SPR118-1101	Vessel
SPR118-1102	Plug, Bottom
SPR118-1103	Plug, Top
SPR118-1104	Diaphragm, Sample
SPR118-1105	Ring, Diaphragm
SPR118-1106	Piston, Sample Expansion/Shrinkage
SPR118-1107	Rod, LVDT Core
SPR118-1112	Cap, LVDT Housing
SPR118-1114	Housing Assembly, LVDT
SPR118-1124	Spacer, Insulating, Housing
SPR118-PN001	Interface, DIN Rail, LVDT
SPR118-PN002	Coil, LVDT
SPR118-PN003	Core, LVDT
SPR118-PN004	Interface, Modbus Interface
SPR118-PN005	Interface, Analog Input, 2 channel
SPR118-PN007	Interface, Bus Termination
SPR118-PN008	Interface, USB/RS485
SPR118-PN009	Cable, Intensifier Power
SPR118-PN010	Cable, LVDT Transducer
SPR118-PN011	Cable, RJ45 LAN
SPR118-PN012	Adapter, RJ45/DB9
SPR118-PN013	Transducer, Pressure
SPR118-PN014	Power Supply, DIN Rail, +24vdc, Universal Input
SPR118-PN015	Spring, LVDT Housing
SPR118-PN016	Tube, SS, High Pressure
SPR118-PN017	Cap, SS, High Pressure Tube
SPR118-PN018	Adapter, SS, High Pressure – 1/8"NPT
SPR118-PN019	O-ring, Piston
SPR118-PN020	Interface, USB/RS232
SPR118-PN021	Seal Kit, Intensifier
SPR118-PN022	Adapter, RJ45/DB25
C08725	Cable, RS232, 25 foot
348-1104	Ring, Backup, High Pressure Seal
80-0021	Thermocouple, Plug
C08564	Retaining Ring, Plug
C08565	O-ring, Viton, Plug
7750-0115	Handle, Top Plug

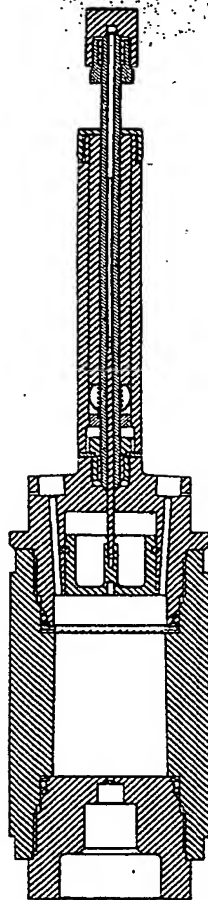
To ensure correct part replacement, always specify model and serial number of instrument when ordering or corresponding.

Section 6 - Drawings and Schematics

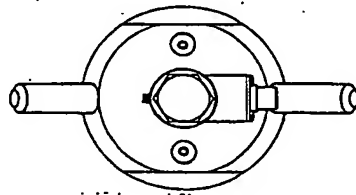
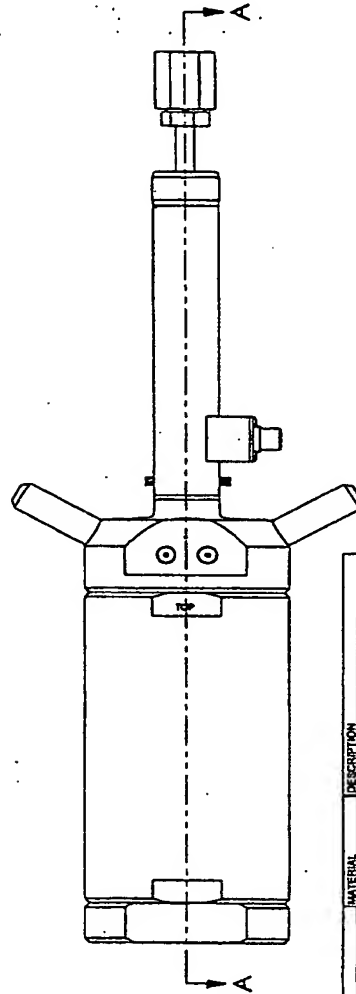
Drawing Number	Description
SPR118-1000	Vessel Assembly
SPR118-1030	Diagram, System
SPR118-1031	Diagram, Tubing
SPR118-1032	Diagram, Wiring, SGSA
SPR118-1033	Diagram, Serial Communication
SPR118-1061	Specification - Setup – Temperature Controller
SPR118-1062	Specification - Setup – Pressure Controller
348-1032	Tubing Diagram, Pressure Control System
348-1033	Wiring Diagram, Pressure Control System

- NOTES:
1. MAXIMUM
 2. MAXIMUM
 3. VESSEL IN
 4. VOLUME A
 5. LVOT MEA
 6. VESSEL A:

RESSURE: 10,000 PSIG (69.1 MPa)
 EMPTERATURE: 400 °F
 ERNAL VOLUME: 200mL BELOW DIAPHRAGM.
 ASUREMENT RANGE: ±0.500 INCH (±12.7mm)
 REMBLY MUST BE PRESSURE TESTED TO 15,000 PSIG, ROOM TEMPERATURE.



SECTION A-A

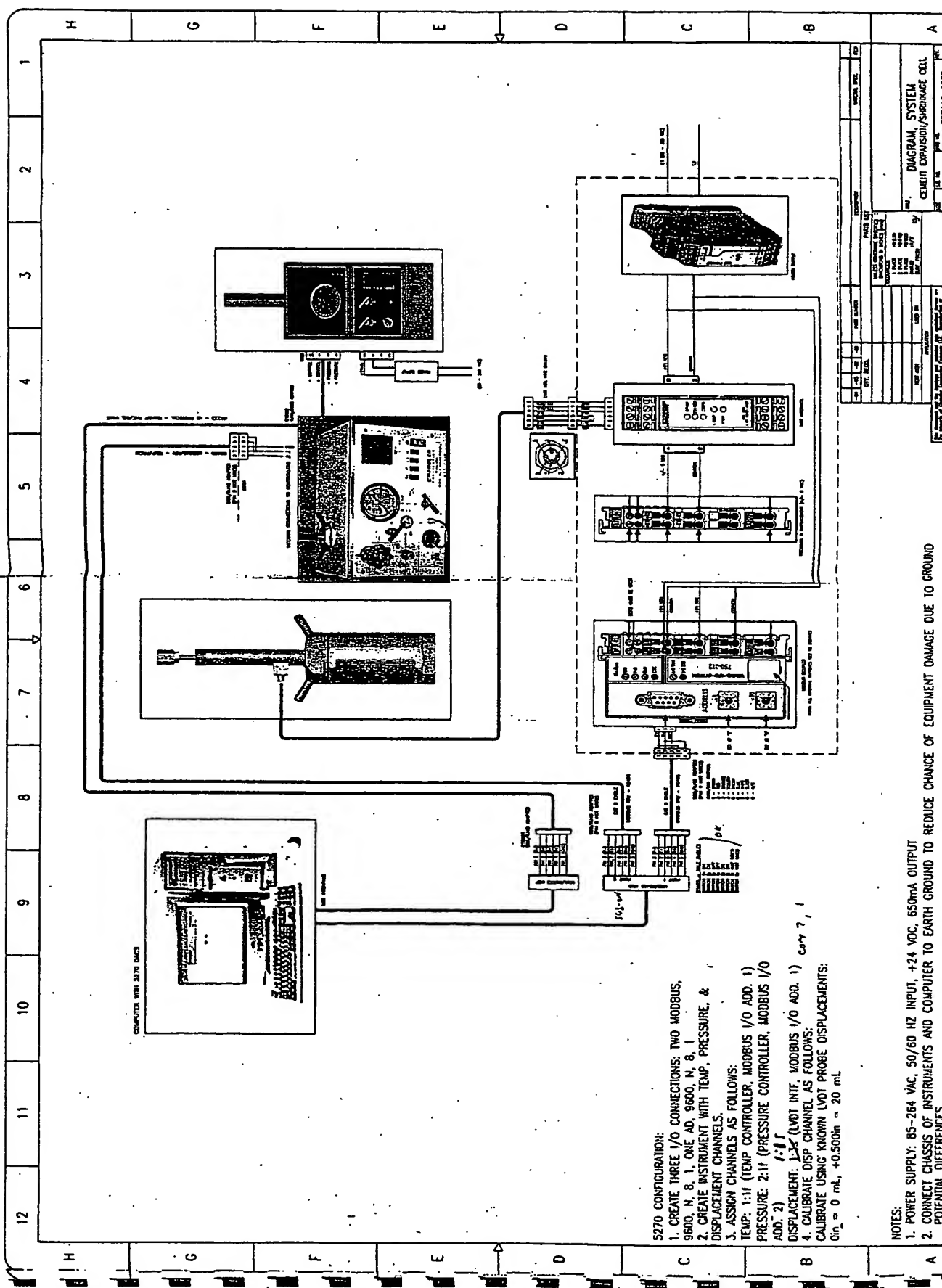


ITEM NO.	QTY	PA	ITEM NO.	QTY	PA
18-101	1	SP	18-101	1	SP
18-102	2	SP	18-102	2	SP
18-103	3	SP	18-103	3	SP
18-104	4	SP	18-104	4	SP
18-105	5	SP	18-105	5	SP
18-106	6	SP	18-106	6	SP
18-107	7	SP	18-107	7	SP
18-108	8	SP	18-108	8	SP
18-109	9	SP	18-109	9	SP
18-110	10	SP	18-110	10	SP
18-111	11	SP	18-111	11	SP
18-112	12	SP	18-112	12	SP
18-113	13	SP	18-113	13	SP
18-114	14	SP	18-114	14	SP
18-115	15	SP	18-115	15	SP
18-116	16	SP	18-116	16	SP
18-117	17	SP	18-117	17	SP
18-118	18	SP	18-118	18	SP
18-119	19	SP	18-119	19	SP
18-120	20	SP	18-120	20	SP
18-121	21	SP	18-121	21	SP
18-122	22	SP	18-122	22	SP
18-123	23	SP	18-123	23	SP
18-124	24	SP	18-124	24	SP

NO.	MATERIAL	DESCRIPTION
18-101	ALUMINUM	VESSEL
18-102	ALUMINUM	DIAPHRAGM, SAMPLE
18-103	ALUMINUM	RVNO, DIAPHRAGM
18-104	ALUMINUM	PLUG, TOP
18-105	ALUMINUM	PLUG, BOTTOM
18-106	ALUMINUM	PLUG, EXPANSION, SAMPLE
18-107	ALUMINUM	PLUG, EXPANSION, LVOT
18-108	ALUMINUM	PLUG, EXPANSION, LVOT
18-109	ALUMINUM	PLUG, EXPANSION, LVOT
18-110	ALUMINUM	PLUG, EXPANSION, LVOT
18-111	ALUMINUM	PLUG, EXPANSION, LVOT
18-112	ALUMINUM	PLUG, EXPANSION, LVOT
18-113	ALUMINUM	PLUG, EXPANSION, LVOT
18-114	ALUMINUM	PLUG, EXPANSION, LVOT
18-115	ALUMINUM	PLUG, EXPANSION, LVOT
18-116	ALUMINUM	PLUG, EXPANSION, LVOT
18-117	ALUMINUM	PLUG, EXPANSION, LVOT
18-118	ALUMINUM	PLUG, EXPANSION, LVOT
18-119	ALUMINUM	PLUG, EXPANSION, LVOT
18-120	ALUMINUM	PLUG, EXPANSION, LVOT
18-121	ALUMINUM	PLUG, EXPANSION, LVOT
18-122	ALUMINUM	PLUG, EXPANSION, LVOT
18-123	ALUMINUM	PLUG, EXPANSION, LVOT
18-124	ALUMINUM	PLUG, EXPANSION, LVOT

ITEM NO.	QTY	PA	ITEM NO.	QTY	PA
18-101	1	SP	18-101	1	SP
18-102	2	SP	18-102	2	SP
18-103	3	SP	18-103	3	SP
18-104	4	SP	18-104	4	SP
18-105	5	SP	18-105	5	SP
18-106	6	SP	18-106	6	SP
18-107	7	SP	18-107	7	SP
18-108	8	SP	18-108	8	SP
18-109	9	SP	18-109	9	SP
18-110	10	SP	18-110	10	SP
18-111	11	SP	18-111	11	SP
18-112	12	SP	18-112	12	SP
18-113	13	SP	18-113	13	SP
18-114	14	SP	18-114	14	SP
18-115	15	SP	18-115	15	SP
18-116	16	SP	18-116	16	SP
18-117	17	SP	18-117	17	SP
18-118	18	SP	18-118	18	SP
18-119	19	SP	18-119	19	SP
18-120	20	SP	18-120	20	SP
18-121	21	SP	18-121	21	SP
18-122	22	SP	18-122	22	SP
18-123	23	SP	18-123	23	SP
18-124	24	SP	18-124	24	SP

THIS DOCUMENT CONTAINS UNCLASSIFIED INFORMATION

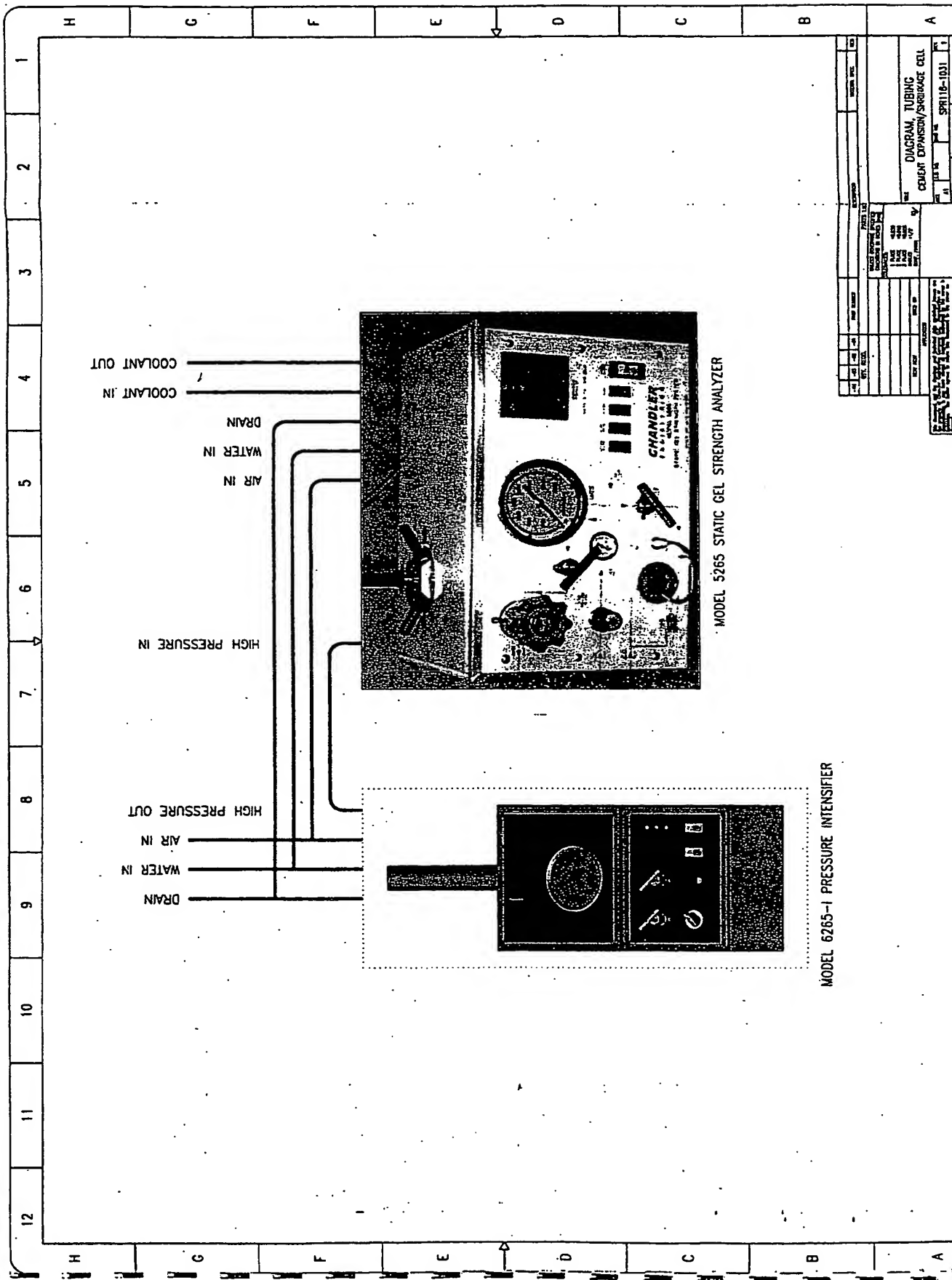


5270 CONFIGURATION:
 1. CREATE THREE I/O CONNECTIONS: TWO MODBUS, 9600, N, 8, 1, ONE AD, 9600, N, 8, 1
 2. CREATE INSTRUMENT WITH TEMP, PRESSURE, & DISPLACEMENT CHANNELS.
 3. ASSIGN CHANNELS AS FOLLOWS:
 TEMP: 1:11 (TEMP CONTROLLER, MODBUS I/O ADD. 1)
 PRESSURE: 2:11 (PRESSURE CONTROLLER, MODBUS I/O ADD. 2)
 DISPLACEMENT: 1:34 (LVDT INIT, MODBUS I/O ADD. 1) *copy 7, 1*
 4. CALIBRATE DISP CHANNEL AS FOLLOWS:
 CALIBRATE USING KNOWN LVDT PROBE DISPLACEMENTS:
 0in = 0 mV, +0.500in = 20 mV

- NOTES:
1. POWER SUPPLY: 85-264 VAC, 50/60 HZ INPUT, +24 VDC, 650mA OUTPUT
 2. CONNECT CHASSIS OF INSTRUMENTS AND COMPUTER TO EARTH GROUND TO REDUCE CHANCE OF EQUIPMENT DAMAGE DUE TO GROUND POTENTIAL DIFFERENCES.

PARTS LIST		QUANTITY		DESCRIPTION	
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1
11	1	1	1	1	1
12	1	1	1	1	1

DIAGRAM SYSTEM
 CEMENT EXPANSION/SHRINKAGE CELL



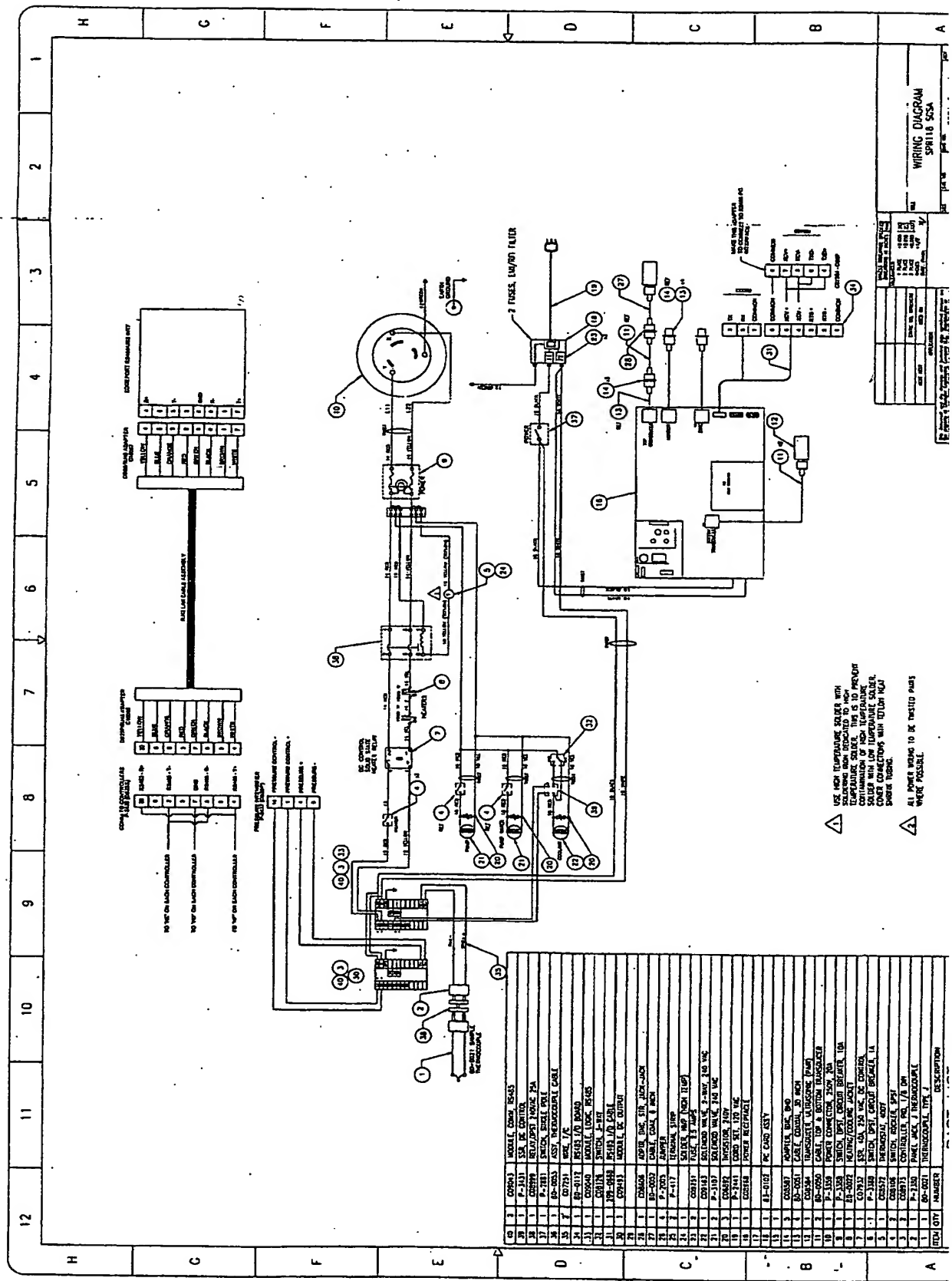
MODEL 6265-I PRESSURE INTENSIFIER

MODEL 5265 STATIC GEL STRENGTH ANALYZER

PART 12		PART 13	
NO. 12	NO. 13	NO. 14	NO. 15
1	2	3	4
5	6	7	8
9	10	11	12
13	14	15	16
17	18	19	20
21	22	23	24
25	26	27	28
29	30	31	32
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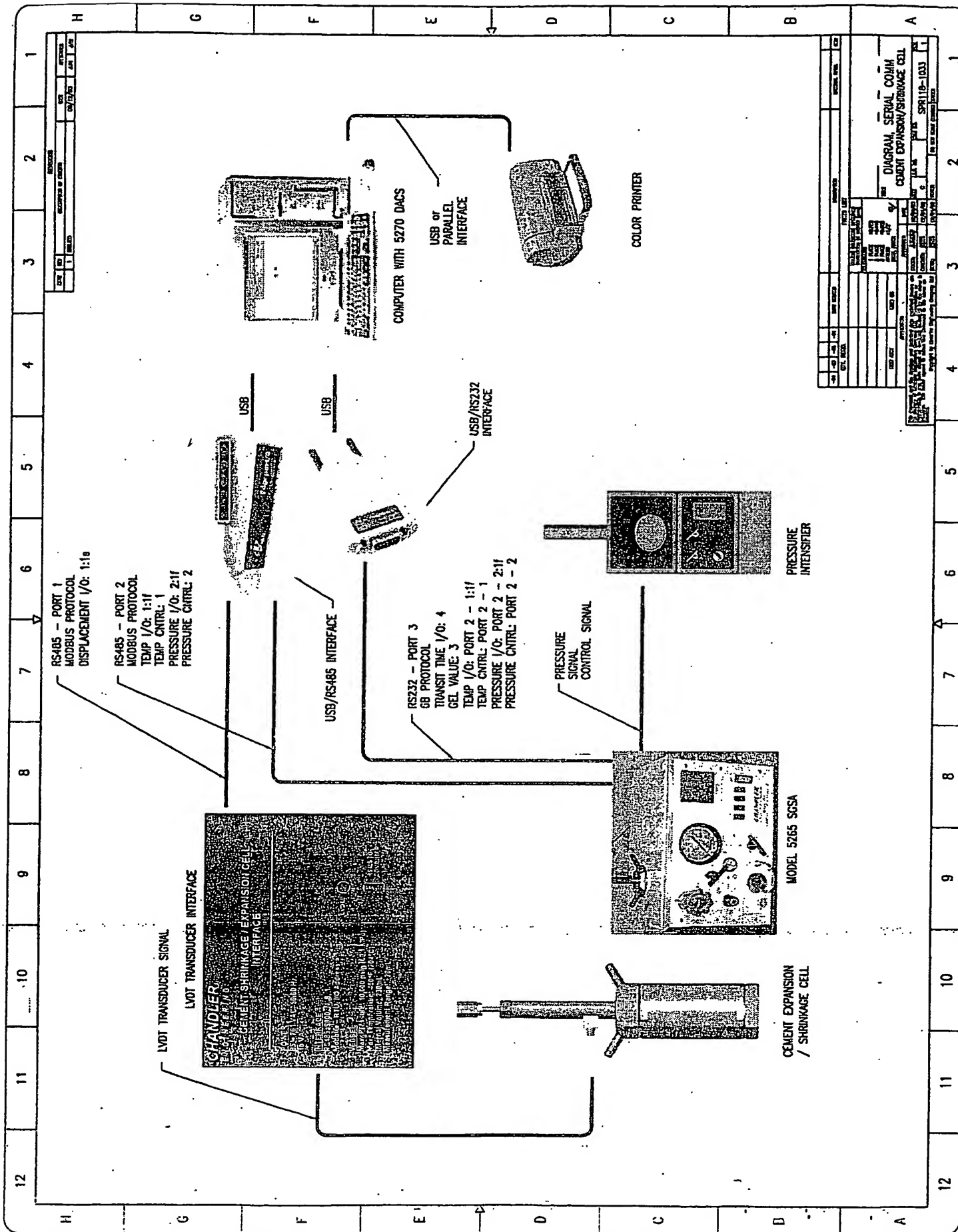
DIAGRAM, TUBING
CONTENT EXPANSION/SHRINKAGE CELL

SPR116-1031

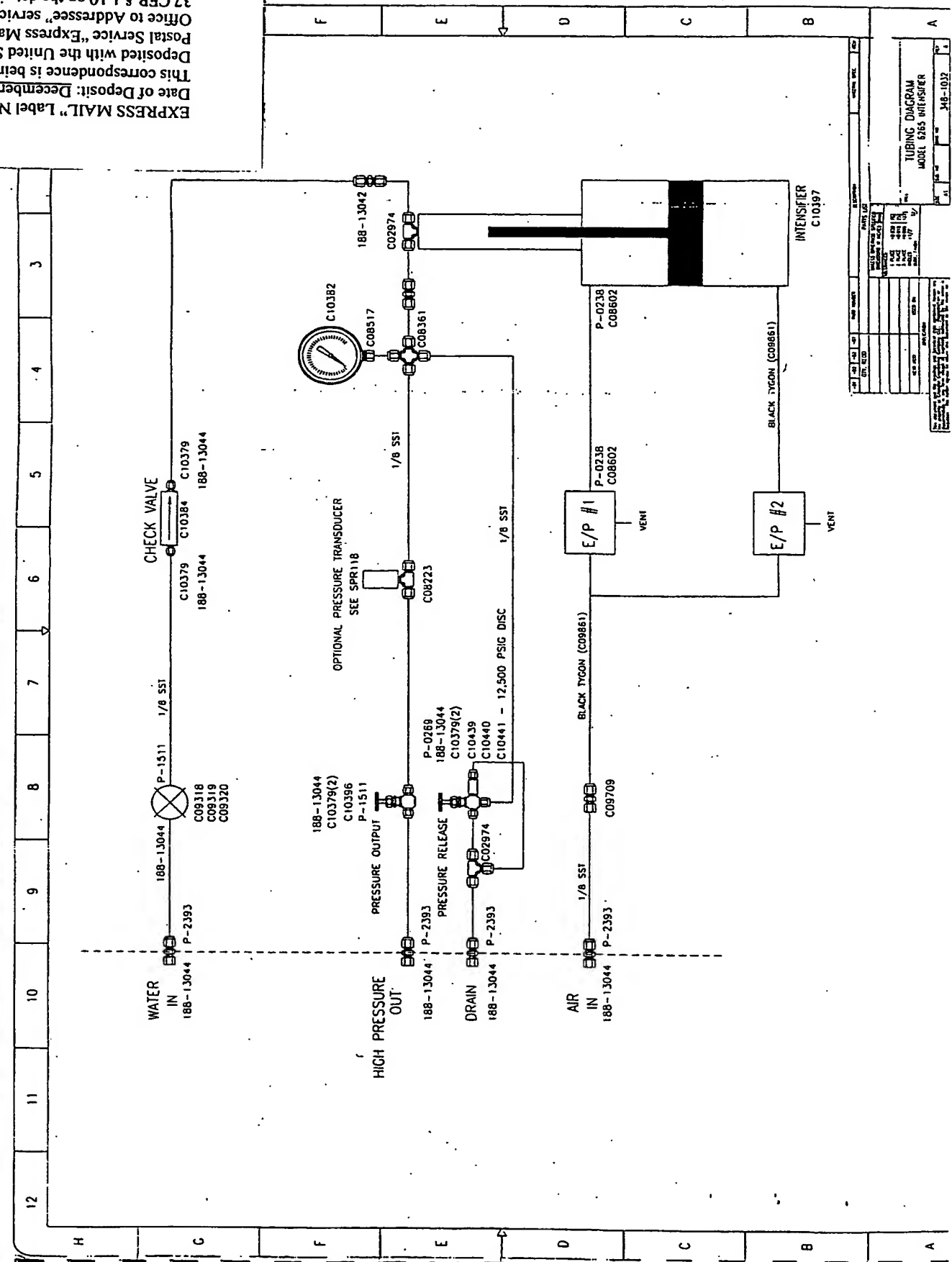


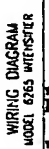
USE HIGH TEMPERATURE SOLDER WITH
TENSILE STRENGTH OF 100 PSI.
CONTAMINATION OF HIGH TEMPERATURE
SOLDER WITH LOW TEMPERATURE SOLDER
WILL CAUSE CRACKS WITH TIGHT Joints.
SOLDER TUBES.

ALL POWER WIRING TO BE TESTED FIRST
WHERE POSSIBLE.



EXPRESS MAIL Label No.: ET537601231US
 Date of Deposit: December 31, 2003
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 Postal Service "Express Mail Post
 Office to Addressee" service under
 37 CFR § 1.10 on the date indicated
 above and addressed to:
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, VA 22313-1450





TITLE: Specification - Setup -Temperature Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Revision	Date	Revised By	Description	Checked By
1	06/08/2003	JJM	Pre-Release	BD

Configuration

Please refer to the following illustration for the location of the electrical connections to the controller:

Controller Electrical Connections		
Terminal	Description	Comment
L	Power input – L1	85 – 264 VAC, 50/60 Hz
N	Power input – H (L2)	
G	Chassis ground	
V+	Input (+)	From "J" Thermocouple
V-	Input (-)	From "J" Thermocouple (Red)
HD	Comm1 – digital ground	
HE	Comm1 – A+ (RS485)	
HF	Comm1 – B- (RS485)	
1A	DC output (+)	Logic – used to control heater SSR
1B	DC output (-)	Signal Ground
2A	Alarm contact	
2B	Alarm contact common	
LA	Digital input	
LB	Digital input	
LC	Digital input common	

Controller Configuration Parameters			
Parameter	Description	Status	Value
RUN	Program Run List	Hide	
PROG	Program Edit List	Altr	
Hb	Holdback type	Hide	OFF
Hb.U	Holdback units	Hide	0
rmP.U	Ramp units	Hide	min
dwL.U	Dwell units	Hide	min
CYC.n	Number of program cycles	Hide	1
SEG.ii	Segment number		varies depending on segment being configured
TYPE	Segment type		varies depending on segment being configured
AL	Alarm List	Hide	
ATUN	Autotune List	Hide	
PID	PID List	Hide	
GSP	Not used		
SET	PID1 or PID2		PID1
PB	Prop. Band		12
TI	Int. Time		400
TD	Der. Time		85
RES	Manual Reset		0
HCB	Cutback High		Auto
LCB	Cutback Low		Auto
SP	Setpoint List	Hide	
SSEL	Setpoint Select		SP1

TITLE: Specification - Setup -Temperature Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Controller Configuration Parameters			
Parameter	Description	Status	Value
<u>SP 1</u>	Setpoint 1 value		0
<u>SP 2</u>	not used		not used
<u>SP L</u>	SP1 low limit		0
<u>SP H</u>	SP1 high limit		400°F (204.4°C)
<u>SP2 L</u>	not used		not used
<u>SP2 H</u>	not used		not used
<u>SPrr</u>	Ramp rate value		Off
<u>Hb.ty</u>	Holdback type		Off
<u>IP</u>	Input List	Hide	
<u>Flt</u>	Filter time constant		1.0
<u>OFS.1</u>			0
<u>Li.2</u>	not used		
<u>Hi.1</u>	not used		
<u>F.1</u>	not used		
<u>PU.iP</u>	iP.1		iP.1
<u>CAL</u>	Factory/User Cal		FACT
<u>CAL.S</u>	calibration point		none
<u>ADJ</u>	not used		
<u>OFS.1</u>	IP1 cal offset		0
<u>OFS.2</u>	not used		
<u>mU.1</u>	IP1 meas. Input		0
<u>mU.2</u>	not used		
<u>CJC.1</u>	CJC reading		N/A
<u>CJC.2</u>	not used		
<u>Li.1</u>	IP1 lin. Input		N/A
<u>L1.2</u>	not used		
<u>PU.SL</u>	selected PV input		iP.1
<u>OP</u>	Output List	Hide	
<u>OP.Lo</u>	Low power limit		0
<u>OP.Hi</u>	High power limit		100
<u>OP.rr</u>	Output rate limit		OFF
<u>FOP</u>	Forced output level		0
<u>CYC.H</u>	Heat cycle time		1.0
<u>ont.H</u>			Auto
<u>End.P</u>			0.0
<u>Sb.OP</u>	Sensor break output power		0
<u>CMS</u>	Comms List	Hide	
<u>Addr</u>	Instrument address, if comm. is used		1
<u>INFO</u>	Information List	Hide	
<u>disp</u>	Configure lower readout content		Std
<u>ACCS</u>	Access List		
<u>code</u>	Access password		1
<u>GoTo</u>	Goto level		Oper/Edit/Conf
<u>Conf</u>	Configuration password		2
<u>INST</u>	Instrument Configuration		
<u>Ctrl</u>	Control type		PID
<u>Act</u>	Control action		Rev
<u>Cool</u>	Type of cooling		Lin
<u>Ti.td</u>	Int. & Der. Time units		Sec

TITLE: Specification - Setup -Temperature Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Controller Configuration Parameters			
Parameter	Description	Status	Value
<u>dTyp</u>			PU
<u>m-A</u>	Front panel Auto/Man		EnAb
<u>r-h</u>	Front panel Run/Hold		EnAb
<u>PwrF</u>	Power feedback		On
<u>Fwd.t</u>	Feed forward type		None
<u>Pdtr</u>	Manual/Auto transfer		No
<u>Sbr.t</u>	Sensor break output		Sb.OP
<u>FOP</u>	Forced manual output		Step
<u>bcd</u>	BCD input		None
<u>GSch</u>	Gain schedule		No
PU	Process Value Configuration		
<u>unit</u>	units		°F or °C
<u>dec.P</u>	decimal points		nnnn.
<u>rng.L</u>	Range low		0
<u>rng.H</u>	Range high		400°F (204.4°C)
IP	Input Type		
<u>inPt</u>	Input Type		J.t/c
<u>CJC</u>			Auto
<u>imP</u>	Sensor Break impedance		Off
SP	Setpoint Configuration		
<u>nSP</u>	Number of setpoints		2
<u>rm.tr</u>	Remote track		OFF
<u>m.tr</u>	Manual track		OFF
<u>Pr.tr</u>	Programmer track		OFF
<u>rmP.U</u>	Setpoint rate limit units		Pmin
<u>rml</u>	Remote setpoint conf.		None
AL1/2/3/4	Alarm Configuration		
<u>AL1-4</u>	Alarm n type		Off
<u>Ltch</u>	Latching		No
PROG	Programmer Configuration		
<u>PtyP</u>	Programmer type		1
<u>HbAc</u>	Holdback		Prog
<u>Pwr.F</u>	Power fail recovery		rSet
<u>Srvo</u>	Starting setpoint of a program		to.PU
LA	Digital Input 1 Configuration		
<u>id</u>	Identity		LoG.i
<u>Func</u>	Function of input		None
LB	Digital Input 2 Configuration		
<u>id</u>	Identity		LoG.i
<u>Func</u>	Function of input		None
HA	Comms 1 Configuration		
<u>id</u>	Identity of module		
<u>Func</u>	Function		Modbus
<u>bAud</u>	Baud rate		9600
<u>dELy</u>	Delay		None
<u>PrtY</u>	Comms parity - only Modbus		1
<u>rES</u>	Comms resolution - only		Full

TITLE: Specification - Setup -Temperature Control r
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Control r Configuration Parameters			
Parameter	Description	Status	Value
	Modbus		
1A/B/C	Module 1 Configuration		
<u>id</u>	Identity of module		Log
<u>Func</u>	Function		Heat
<u>VAL.L</u>	Minimum output		0
<u>VAL.H</u>	Maximum output		100
<u>Out.L</u>	Minimum average power		0
<u>Out.H</u>	Maximum average power		100
2A/B/C	Module 2 Configuration		
<u>id</u>	Identity of module		None
<u>Func</u>	Function		
<u>VAL.L</u>	Minimum output		
<u>VAL.H</u>	Maximum output		
<u>Out.L</u>	Minimum average power		
<u>Out.H</u>	Maximum average power		
3A/B/C	Module 3 Configuration		
<u>id</u>	Identity of module		None
<u>Func</u>	Function		
<u>SEnS</u>	Digital output sense		
<u>Alarm 1</u>	Alarm 1 active		
<u>Alarm 2</u>	Alarm 2 active		
<u>Alarm 3</u>	Alarm 3 active		
<u>Alarm 4</u>	Rate of change alarm		
<u>mAn</u>	Controller in manual mode		
<u>Sbr</u>	Sensor break		
<u>SPAn</u>	PV out of range		
<u>Lbr</u>	Loop break		
<u>Ld.F</u>	Load failure alarm		
<u>Tune</u>	Tuning in progress		
<u>dc.F</u>	Voltage output open circuit		
<u>rmt.F</u>	PDSIO related		
<u>Nw.AL</u>	New alarm has occurred		
<u>End</u>	End of program		
<u>Sync</u>	Program sync.		
CAL	Calibration		
PASS	Password Configuration		
<u>ACC.P</u>	Full or Edit level password		1
<u>cnF.P</u>	Configuration password		2

TITLE: Specification - Setup – Pressure Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Revision	Date	Revised By	Description	Checked By
1	06/08/2003	JJM	Pre-Release	BD

Configuration

Please refer to the following illustration for the location of the electrical connections to the controller:

Controller Electrical Connections		
Terminal	Description	Comment
L	Power input – L1	85 – 264 VAC, 50/60 Hz
N	Power input – H (L2)	
G	Chassis ground	
V+	Input (+)	From Pressure Transducer
V-	Input (-)	From Pressure Transducer
HD	Comm1 – digital ground	
HE	Comm1 – A+ (RS485)	
HF	Comm1 – B- (RS485)	
1A	DC output (+)	0-10 Vdc - used to control hydraulic unit pressure intensifier
1B	DC output (-)	Signal Ground
2A	Alarm contact	
2B	Alarm contact common	
LA	Digital input	
LB	Digital input	
LC	Digital input common	

Controller Configuration Parameters			
Parameter	Description	Status	Value
RUN	Program Run List	Hide	
PROG	Program Edit List	Altr	
<u>Hb</u>	Holdback type	Hide	OFF
<u>Hb.U</u>	Holdback units	Hide	0
<u>rmP.U</u>	Ramp units	Hide	min
<u>dwL.U</u>	Dwell units	Hide	min
<u>CYC.n</u>	Number of program cycles	Hide	1
<u>SEG.n</u>	Segment number		varies depending on segment being configured
<u>TYPE</u>	Segment type		varies depending on segment being configured
AL	Alarm List	Hide	
ATUN	Autotune List	Hide	
PID	PID List	Hide	
<u>SET</u>	PID1 or PID2		PID1
<u>PB</u>	Prop. Band		70
<u>TI</u>	Int. Time		3
<u>TD</u>	Der. Time		OFF
<u>RES</u>	Manual Reset		0
<u>HCB</u>	Cutback High		Auto
<u>LCB</u>	Cutback Low		Auto
SP	Setpoint List	Hide	
<u>SSEL</u>	Setpoint Select		SP1
<u>SP 1</u>	Setpoint 1 value		0

TITLE: Specification - Setup – Pressure Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Controller Configuration Parameters			
Parameter	Description	Status	Value
<u>SP 2</u>	not used		not used
<u>SP L</u>	SP1 low limit		0
<u>SP H</u>	SP1 high limit		10.00
<u>SP2 L</u>	not used		not used
<u>SP2 H</u>	not used		not used
<u>SPrr</u>	Ramp rate value		Off
<u>Hb.ty</u>	Holdback type		Off
<u>IP</u>	Input List	Hide	
<u>Flt</u>	Filter time constant		1.0
<u>OFS.1</u>			0
<u>LI.2</u>	not used		
<u>Hi.i</u>	not used		
<u>F.1</u>	not used		
<u>PU.iP</u>	iP.1		iP.1
<u>CAL</u>	Factory/User Cal		FACT
<u>CAL.S</u>	calibration point		none
<u>ADJ</u>	not used		
<u>OFS.1</u>	IP1 cal offset		0
<u>OFS.2</u>	not used		
<u>mU.1</u>	IP1 meas. Input		0
<u>mU.2</u>	not used		
<u>CJC.1</u>	CJC reading		N/A
<u>CJC.2</u>	not used		
<u>LI.1</u>	IP1 lin. Input		N/A
<u>LI.2</u>	not used		
<u>PU.SL</u>	selected PV input		N/A
<u>OP</u>	Output List	Hide	
<u>OP.Lo</u>	Low power limit		0
<u>OP.Hi</u>	High power limit		100
<u>OP.rr</u>	Output rate limit		OFF
<u>FOP</u>	Forced output level		0 1.6
<u>CYC.H</u>	Heat cycle time		0.20
<u>ont.H</u>			Auto
<u>End.P</u>			0.0
<u>Sb.OP</u>	Sensor break output power		0
<u>CMS</u>	Comms List	Hide	
<u>Addr</u>	Instrument address, if comm. is used		2
<u>INFO</u>	Information List	Hide	
<u>disp</u>	Configure lower readout content		Stat
<u>ACCS</u>	Access List		
<u>code</u>	Access password		1
<u>GoTo</u>	Goto level		Oper/Edit/Conf
<u>Conf</u>	Configuration password		2
<u>INST</u>	Instrument Configuration		
<u>Ctrl</u>	Control type		PID
<u>Act</u>	Control action		Rev
<u>Cool</u>	Type of cooling		Lin
<u>Ti.td</u>	Int. & Der. Time units		Sec
<u>dTyp</u>			PU

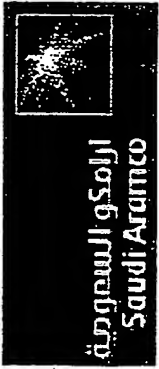
TITLE: Specification - Setup – Pressure Controller
Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Controller Configuration Parameters			
Parameter	Description	Status	Value
<u>m-A</u>	Front panel Auto/Man		EnAb
<u>r-h</u>	Front panel Run/Hold		EnAb
<u>Pwr.F</u>	Power feedback		On
<u>Fwd.t</u>	Feed forward type		None
<u>Pdtr</u>	Manual/Auto transfer		No
<u>Sbr.t</u>	Sensor break output		Sb.OP
<u>FOP</u>	Forced manual output		Step
<u>bcd</u>	BCD input		None
<u>GSch</u>	Gain schedule		No
PU	Process Value Configuration		
<u>unit</u>	units		None
<u>dec.P</u>	decimal points		nn.nn
<u>rng.L</u>	Range low		0
<u>rng.H</u>	Range high		7.50
IP	Input Type		
<u>inPt</u>	Input Type		Volt
<u>imP</u>	Sensor Break impedance		Auto
<u>inP.L</u>	Input value low		0.50
<u>inP.H</u>	Input value high		4.5
<u>VAL.L</u>	Displayed reading low		0
<u>VAL.H</u>	Displayed reading high		10.00
SP	Setpoint Configuration		
<u>nSP</u>	Number of setpoints		2
<u>rm.tr</u>	Remote track		OFF
<u>m.tr</u>	Manual track		OFF
<u>Pr.tr</u>	Programmer track		OFF
<u>rmP.U</u>	Setpoint rate limit units		Pmin
<u>rmI</u>	Remote setpoint conf.		None
AL1/2/3/4	Alarm Configuration		
<u>AL1-4</u>	Alarm n type		Off
<u>Ltch</u>	Latching		No
PROG	Programmer Configuration		
<u>PtyP</u>	Programmer type		1
<u>HbAc</u>	Holdback		Prog
<u>Pwr.F</u>	Power fail recovery		rmP.b
<u>Srvo</u>	Starting setpoint of a program		to.PU
LA	Digital Input 1 Configuration		
<u>id</u>	Identity		LoG.i
<u>Func</u>	Function of input		None
LB	Digital Input 2 Configuration		
<u>id</u>	Identity		LoG.i
<u>Func</u>	Function of input		None
HA	Comms 1 Configuration		
<u>id</u>	Identity of module		
<u>Func</u>	Function		Modbus
<u>bAud</u>	Baud rate		9600
<u>dELy</u>	Delay		None

TITLE: Specification - Setup – Pressure Controller

Instrument Model: Model 4268ES Cement Expansion / Shrinkage Cell

Controller Configuration Parameters			
Parameter	Description	Status	Value
<u>PrY</u>	Comms parity - only Modbus		1
<u>rES</u>	Comms resolution - only Modbus		Full
1A/B/C	Module 1 Configuration		
<u>id</u>	Identity of module		Dc.OP
<u>Func</u>	Function		OP
<u>VAL.L</u>	Minimum output		0
<u>VAL.H</u>	Maximum output		10.0
<u>Unit</u>	Unit of measurement		volt
<u>Out.L</u>	Minimum average power		0
<u>Out.H</u>	Maximum average power		10.0
2A/B/C	Module 2 Configuration		
<u>id</u>	Identity of module		None
<u>Func</u>	Function		
<u>VAL.L</u>	Minimum output		
<u>VAL.H</u>	Maximum output		
<u>Out.L</u>	Minimum average power		
<u>Out.H</u>	Maximum average power		
3A/B/C	Module 3 Configuration		
<u>id</u>	Identity of module		None
<u>Func</u>	Function		
<u>SEnS</u>	Digital output sense		
<u>Alarm 1</u>	Alarm 1 active		
<u>Alarm 2</u>	Alarm 2 active		
<u>Alarm 3</u>	Alarm 3 active		
<u>Alarm 4</u>	Rate of change alarm		
<u>mAn</u>	Controller in manual mode		
<u>Sbr</u>	Sensor break		
<u>SPAn</u>	PV out of range		
<u>Lbr</u>	Loop break		
<u>Ld.F</u>	Load failure alarm		
<u>Tune</u>	Tuning in progress		
<u>dc.F</u>	Voltage output open circuit		
<u>rmt.F</u>	PDSIO related		
<u>Nw.AL</u>	New alarm has occurred		
<u>End</u>	End of program		
<u>Sync</u>	Program sync.		
CAL	Calibration		
PASS	Password Configuration		
<u>ACC.P</u>	Full or Edit level password		1
<u>cnF.P</u>	Configuration password		2



R & D Center

File Name: 6761-01-01,74F&100psi

Instrument: 4288ES #101

Lab Technician:

Job Type:

Well ID: 6761-01-01**HRDH-664

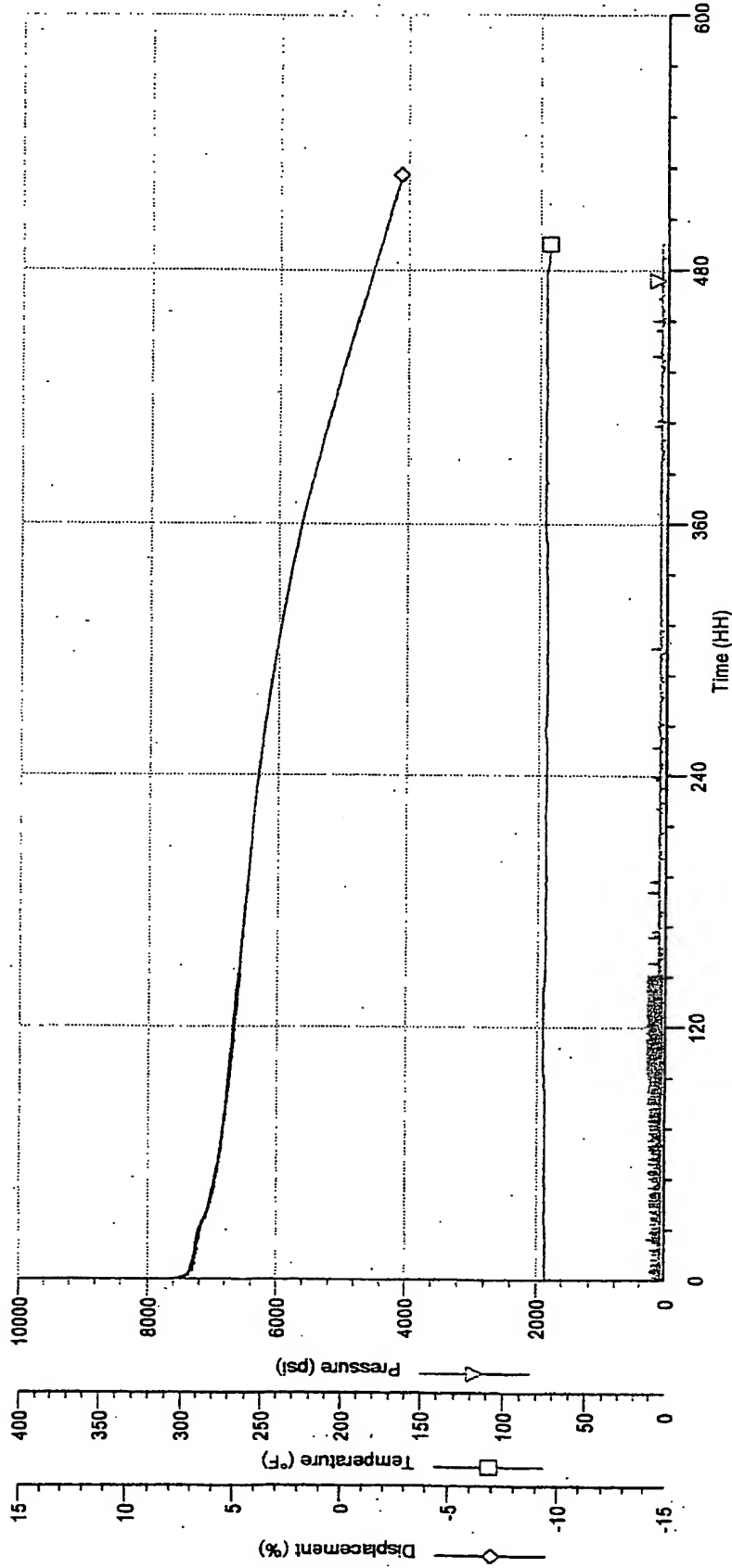
BHST: 74F

BHCT: 74F

Density: 72pcf

Initial Displacement: 7.64 %

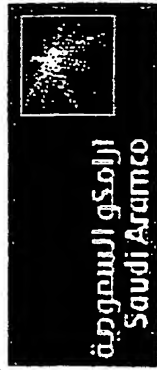
Final Displacement: N/A



Additives: 0.15%D167+0.2%D153+0.06gpsD80+0.025gpsD81

Comments:

Test Stop:
Test Length:

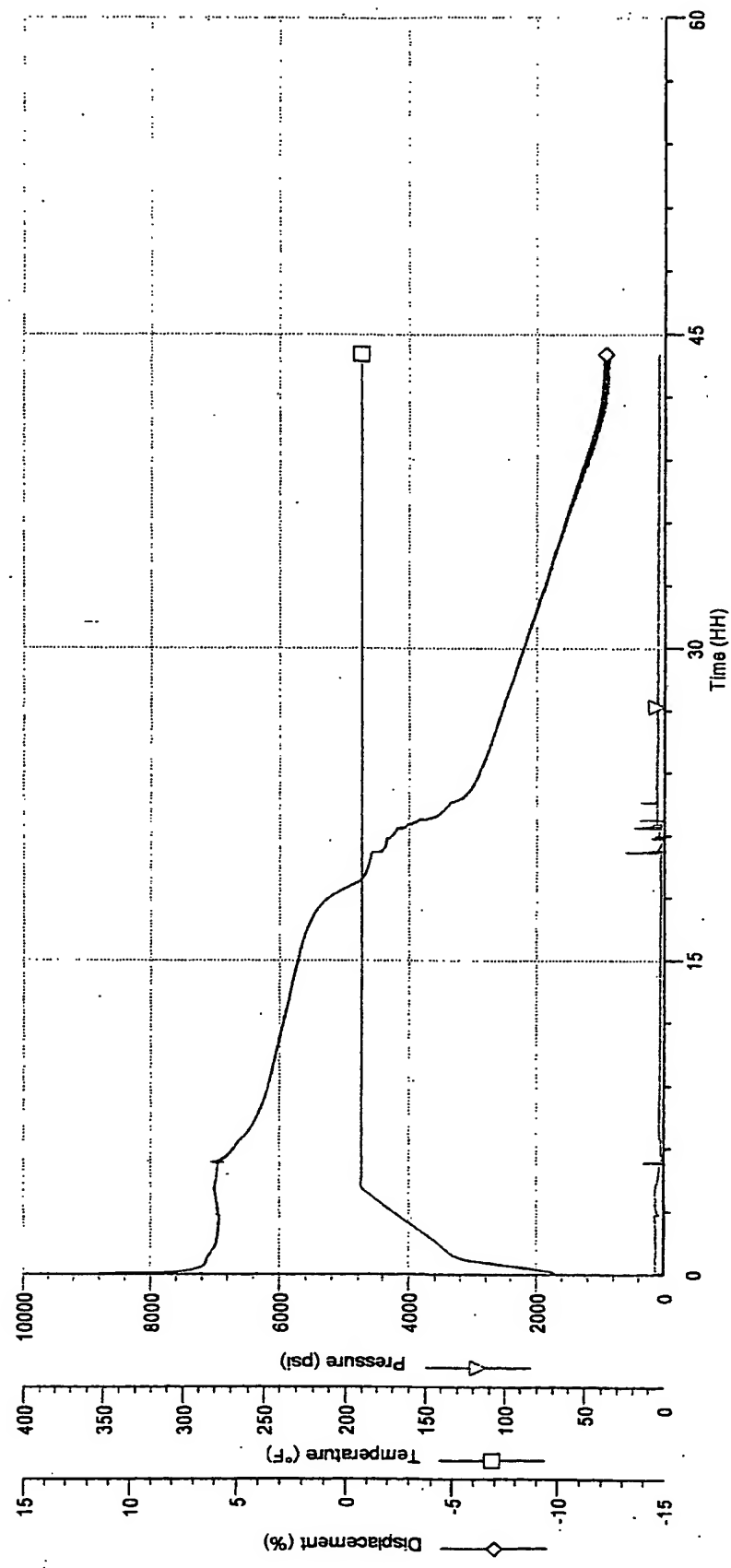


R & D Center

File Name: 6761-01-01
Instrument: 4268ES #101
Lab Technician:
Job Type: 18 5/8"CSG 1st L@9058'

Well ID: 6761-01-01**HRDH-664
BHST: 190F
BHCT: 129F
Density: 71 pcf

Initial Displacement: 11.37 %
Final Displacement: -12.20 %



Additives: 0.15%D167+0.2%D153+0.08gpsD80+0.025gpsD81

Comments: lab mix



R & D Center

File Name: 6344-03-01 101 PCFa

Instrument: 4268ES #101

Lab Technician:

Job Type: 18 5/8"CSG 1st L@9058'

Well ID: 6344-03-01**HRDH-639

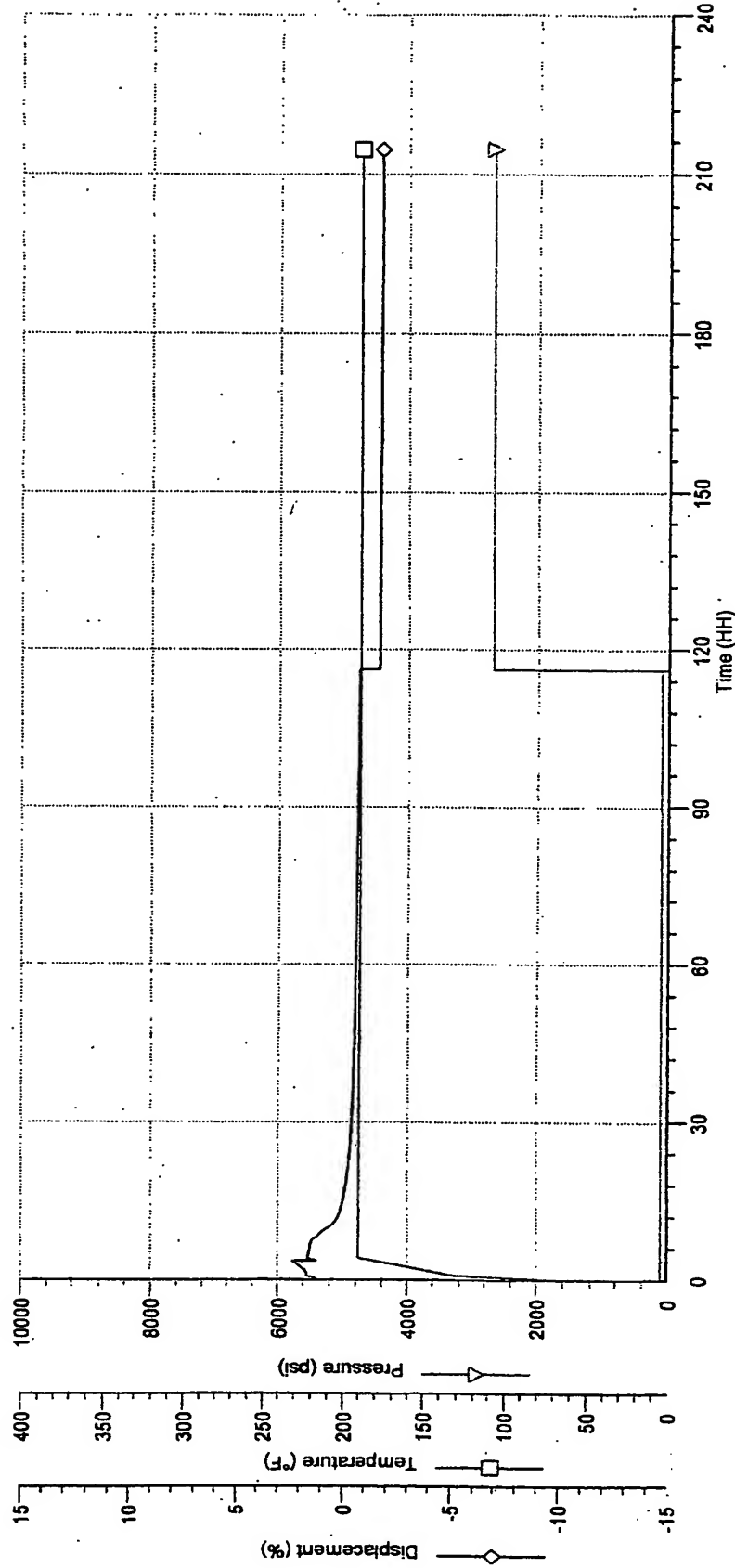
BHST: 190F

BHCT: 129F

Density: 101pcf

Initial Displacement: 1.28 %

Final Displacement: -1.74 %



Additives: 0.03gpsD80+0.6%D112+0.025gpsD81+1.9%Bentonite

Comments:

Test Start:

Test Stop:

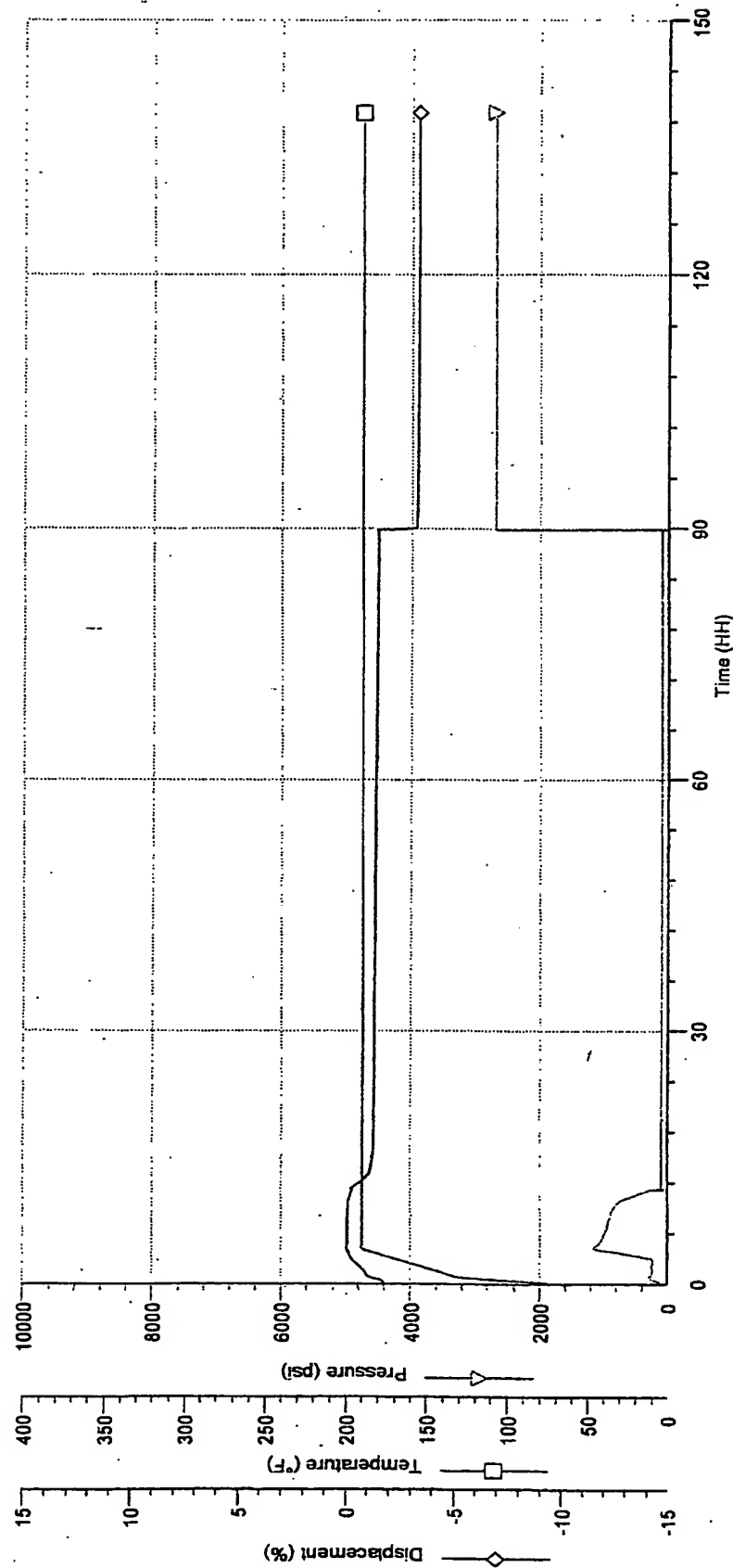


R & D Center

File Name: 6344-03-02, 100psi
Instrument: 4288ES #101
Lab Technician:
Job Type: 9 5/8" CSG 1stT @9162'

Well ID: 6344-03-02**HRDH-639
BHST: 190F
BHCT: 129F
Density: 118pcf

Initial Displacement: -1.71 %
Final Displacement: -3.36 %



Additives: 0.1gpsD80+0.6%D60+0.03gpsD81

Comments: lab blend



R & D Center

File Name: 6344-03-02
Instrument: 4268FS #1n1
Lab Technician:
Job Type: 9 5/8"CSG 1stT@9162'

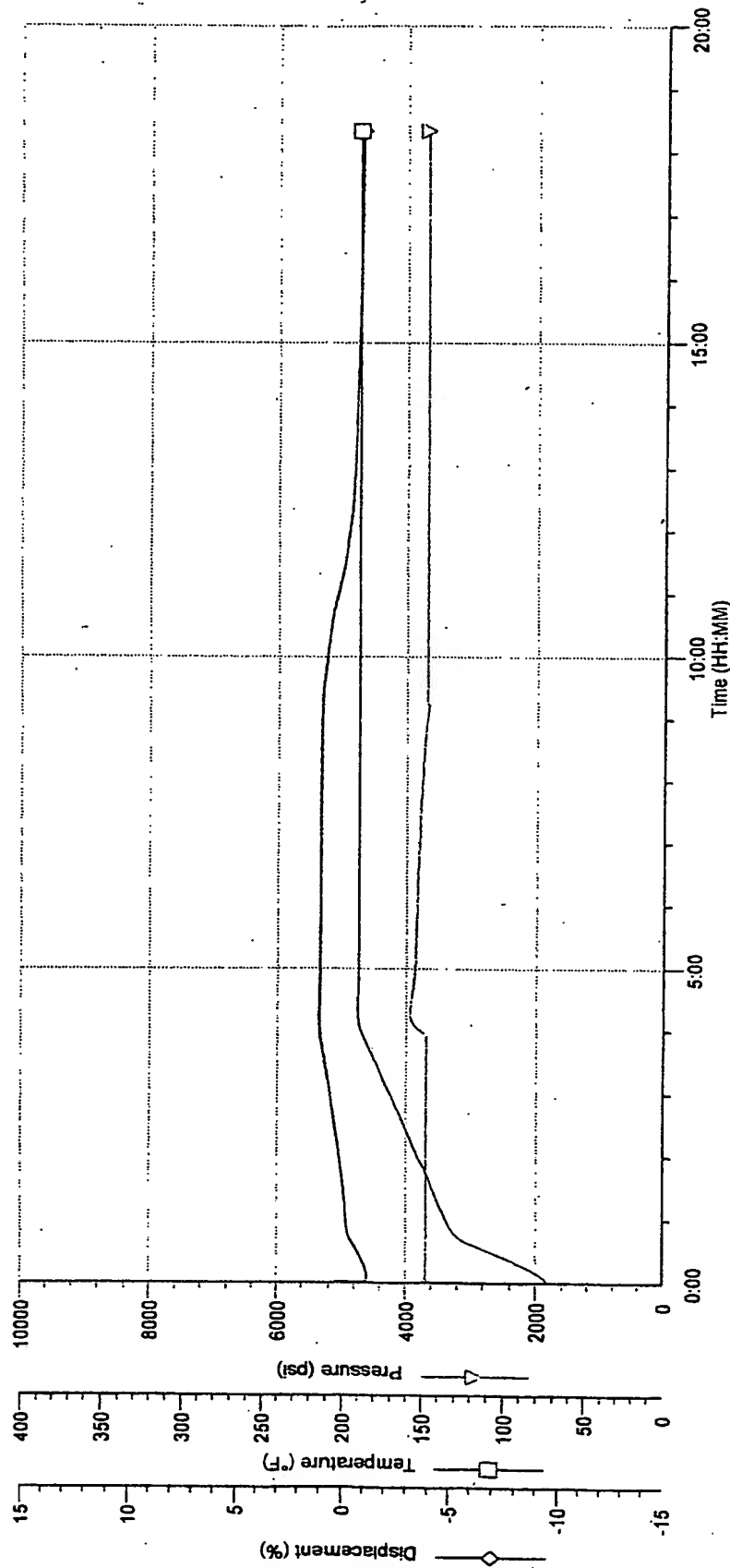
Well ID: 6344-03-02**HRDH-639

BHST: 190F

BHCT: 129F

Density: 118pcf

Initial Displacement: 0.297 %
Final Displacement: -0.899 %



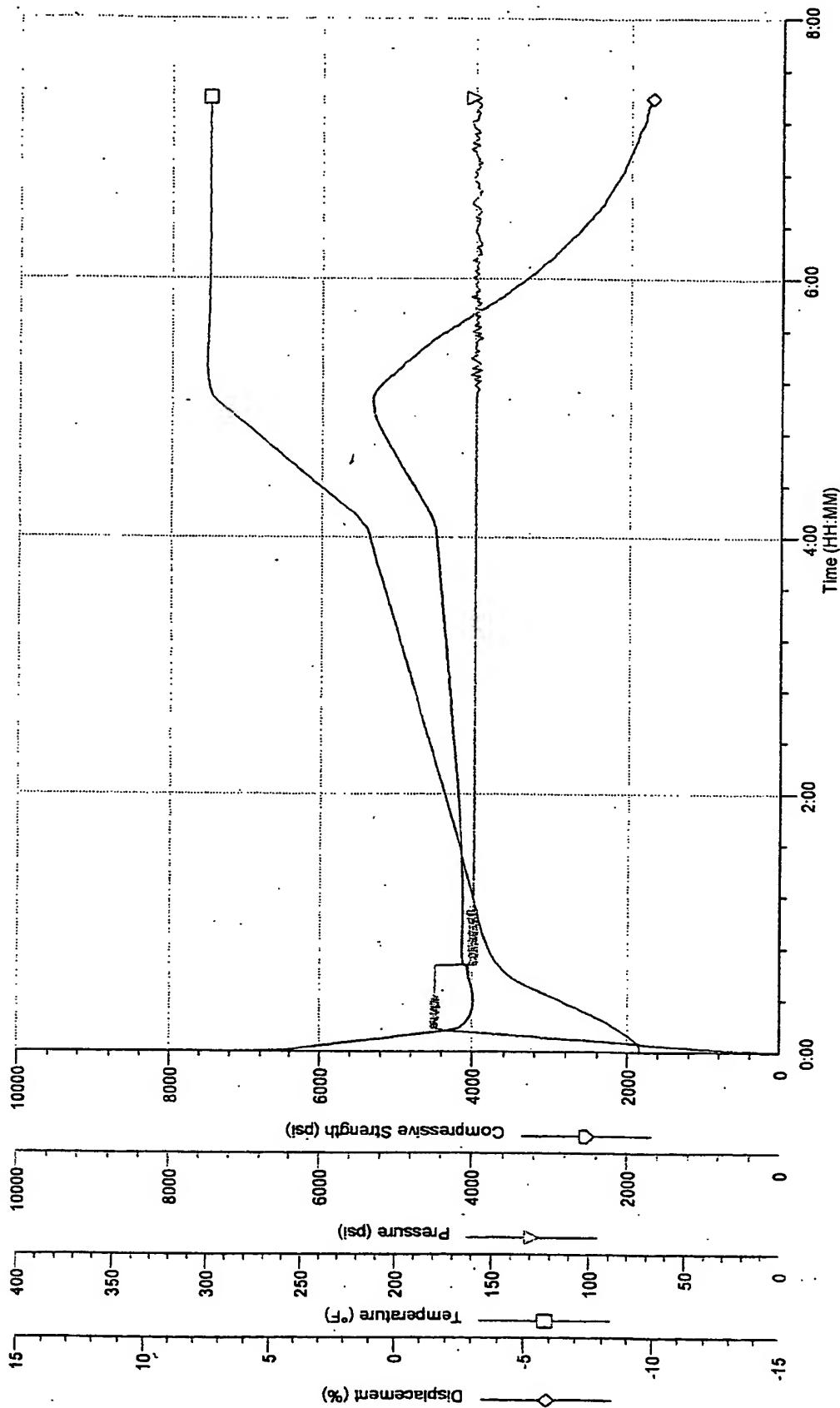
Additives: 0.1gpsD80+0.6%D60+0.03gpsD81

Comments: LAB BLEND

Temperature: 300 °F
Pressure: 4025 psi
Transit Time: ERROR
Zero Gel @ N/A

Well ID: 6761-01-01**HRDH-664
Job Type:
Compressive strength type B (more than 14 lb/gal)

50 psi @ N/A
500 psi @ N/A
Strength: N/A
Final Gel @ N/A



Additives: 0.15%D167+0.2%D153+0.06gpsD80+0.025gpsD81

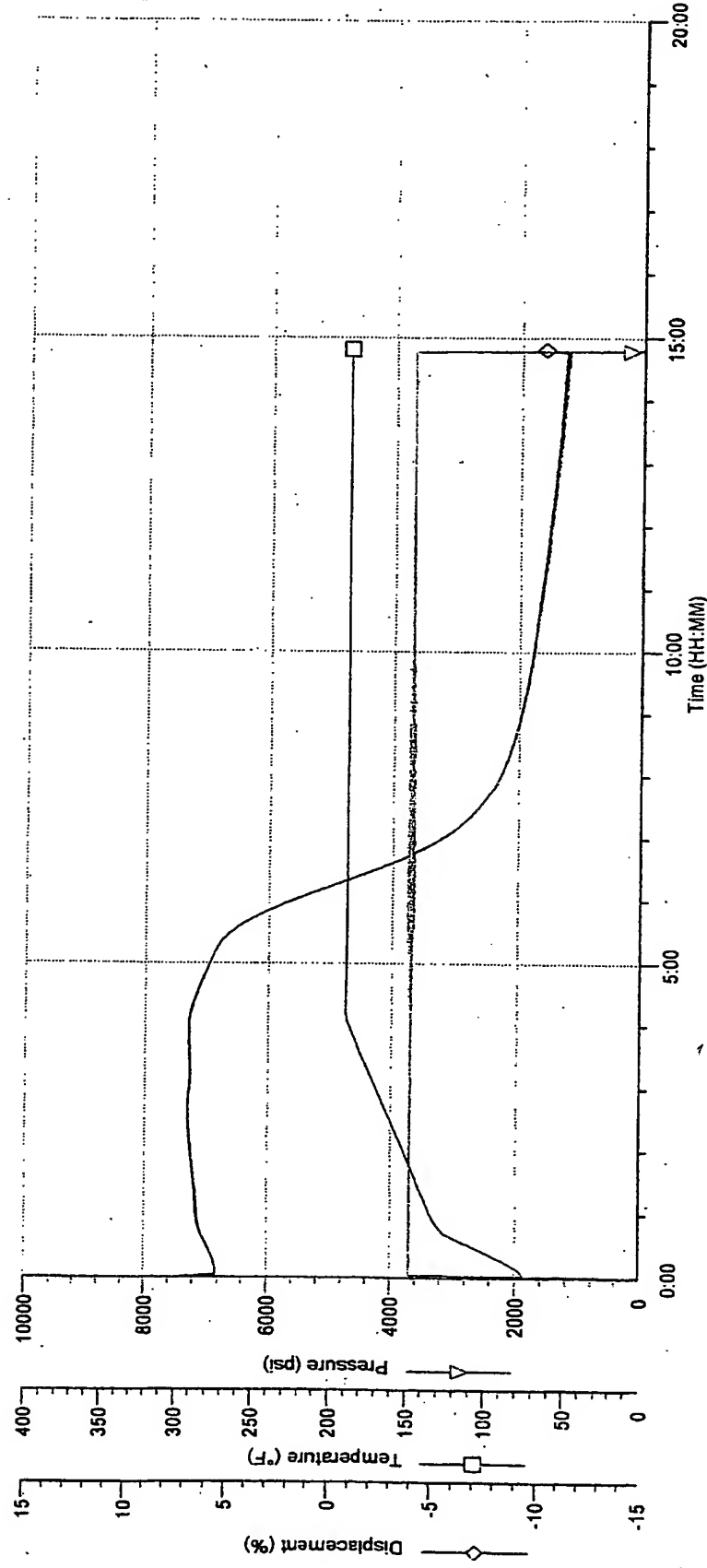
Comments: lab mix not pressurized



File Name: 6855-01-04
Instrument: 4268ES #101
Lab Technician:
Job Type: casing

Well ID: LiteCrete Blend
BHST: 190 Deg F
BHCT:
Density: 71 pcf

Initial Displacement: 7.13 %
Final Displacement: -10.17 %



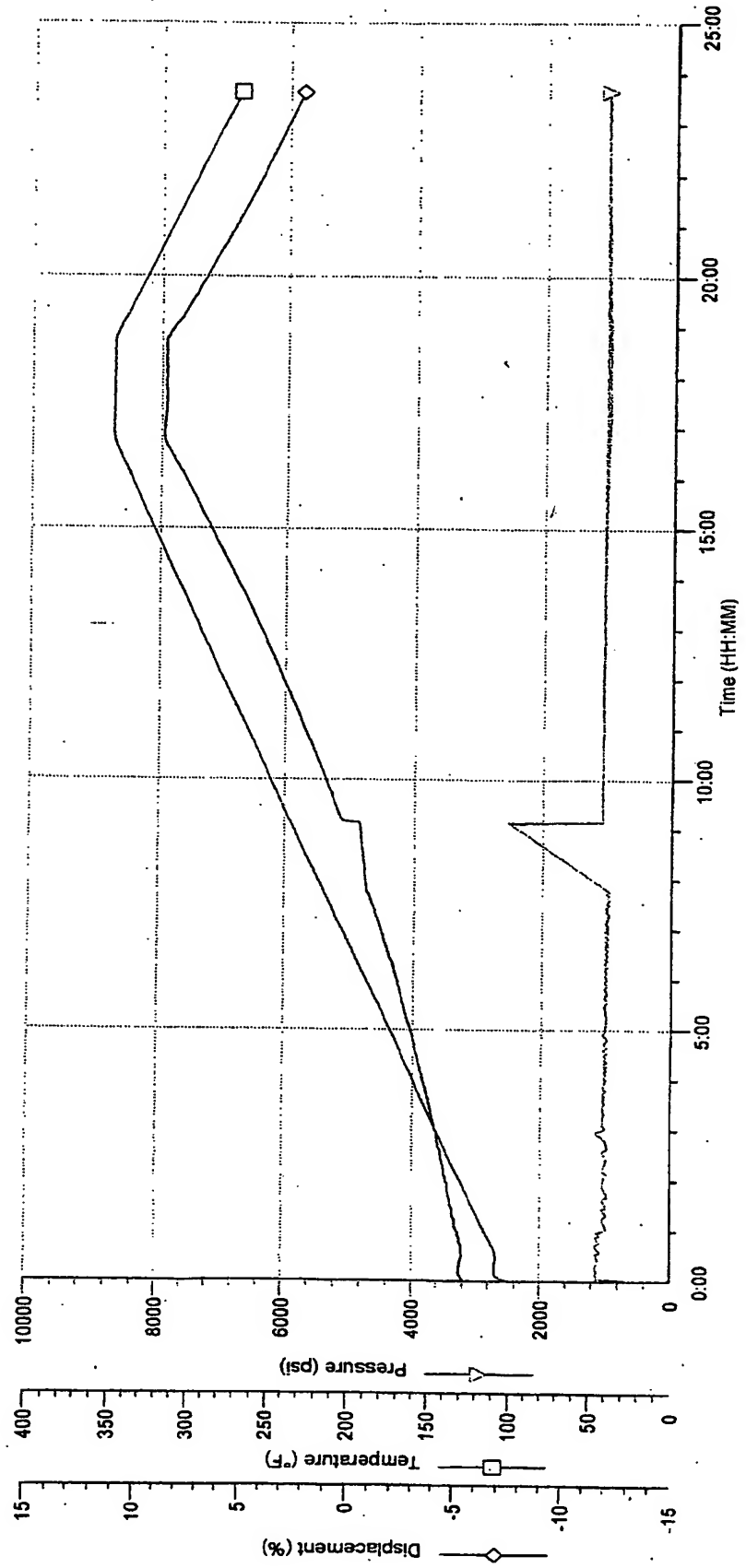
Additives: Dowell

Comments: sample was pressurized to 4200 psi in a HP/HT Consistometer prior to placing in the expansion cell. Total Shrinkage after initial pressurization is 17% for 14:49 (hrs:Mins)



File Name: expansion of water-3
Instrument: 4268ES #101
Lab Technician:
Job Type:

Well ID:
BHST:
BHCT:
Density:
Initial Displacement: -5.39 %
Final Displacement: 2.34 %



Additives:

Comments: Expansion of water to 350 deg F

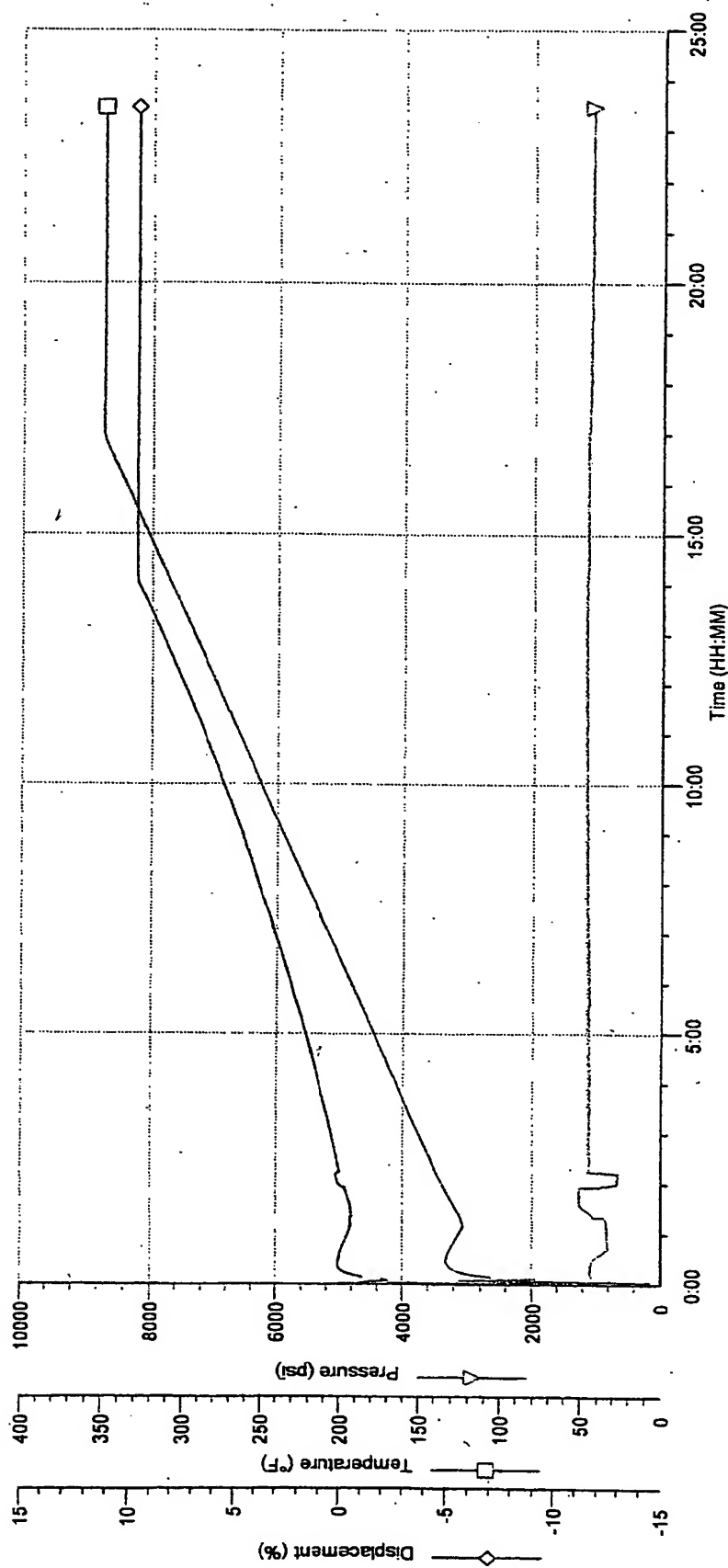


R & D Center

File Name: expansion of water
Instrument: 4288ES #101
Lab Technician:
Job Type:

Well ID:
BHST:
BHCT:
Density:

Initial Displacement: -0.81 %
Final Displacement: 9.68 %



Additives:

Comments:



R & D Center

File Name: 6344-03-01 101 PCF
Instrument: 4268ES #101
Lab Technician:
Job Type: 9 5/8"CSG

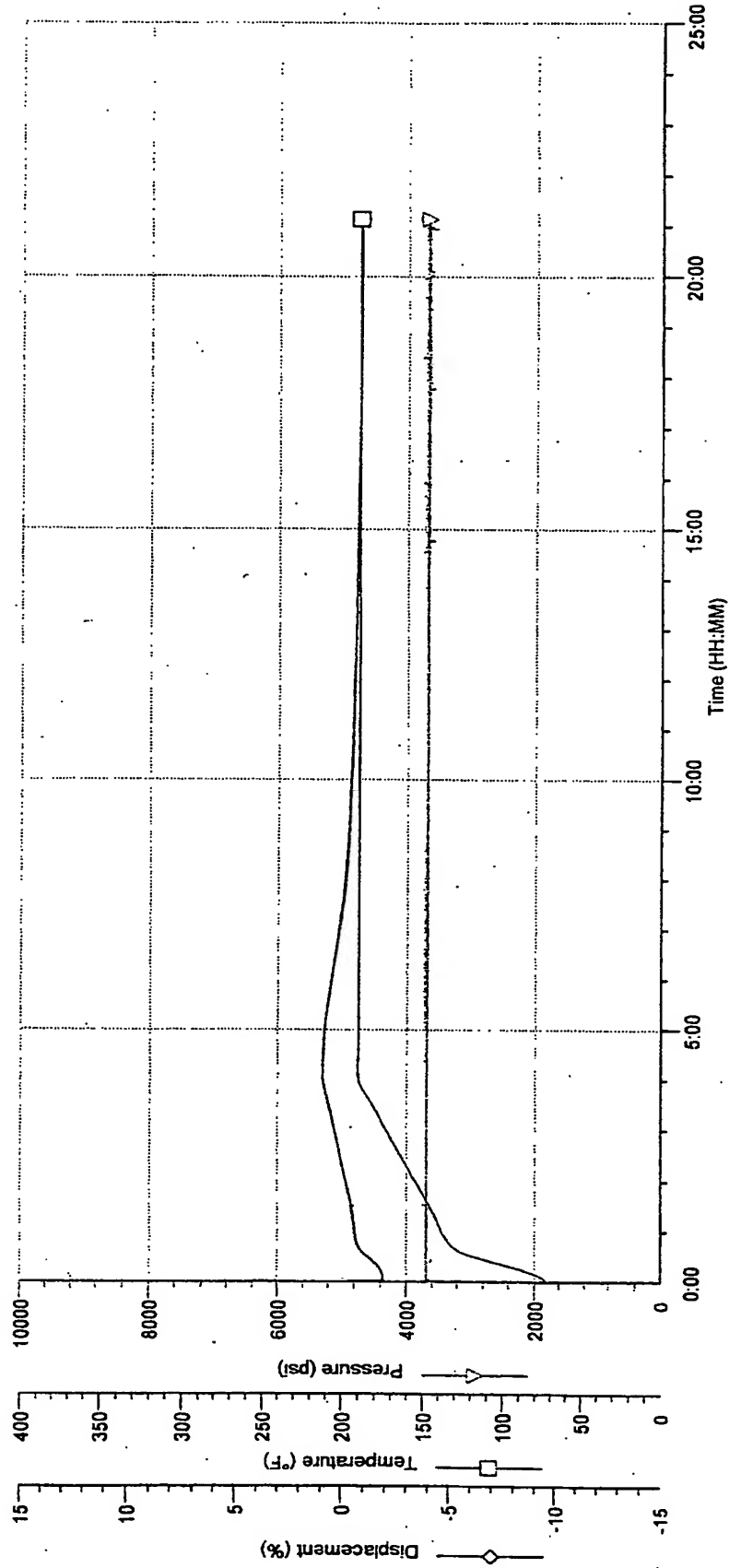
Well ID: 6344-03-01**HRDH-639

BHST: 190F

BHCT: 129F

Density: 101pcf

Initial Displacement: -0.311 %
Final Displacement: -0.824 %



Additives: 0.03gpsD80+0.6%D112+0.025gpsD81

Comments:

Shrinkage & Slurry Density Tests

Date: 01-31-2000

Project no. 98-00752

Cement blend Sample Received 1/15/2000

Hydrostaic pressure @ 6600 ft 11.5 PPG

Surface pump pressure

Test pressure

Test Temperature

3940 psi

1000 psi

4940 psi

140 deg F

Shrinkage Determination	LightCrete Blend cell 1	LightCrete Blend cell 2
% Shrinkage		
initial height	11.25	11.25
reduced height	0.984	1.077
Average of two samples	1.031	
	%volume reduction	
	8.75	9.57
Average of two samples	9.16	

Pressurized Mud Balance Density Determinations		
Slurry Density	Test 1	Test 2
Measured at surface	PCF	PCF
calculated (downhole pressure)	78.5	78.5
measured (downhole pressure)	86	86
	93	94

7 Determination of bulk shrinkage or expansion under impermeable conditions at elevated temperature and pressure.

7.1 General information

The purpose of this test method is to measure the bulk expansion or bulk shrinkage at temperature and pressure. This procedure shall be used when procedure 6 cannot be used for instance in case of high temperature or with compressible slurries (slurries that contain gas, spheres that break under pressure or other pressure/temperature sensitive materials). The method described below is limited by the equipment's temperature and pressure limitations.

7.2 Apparatus

7.2.1 Membrane

The slurry shall be placed in an impermeable flexible container, which is sealed by a lid. The material of membrane must not react in high pH environment, temperature resistant and should be capable to contain at least 500 ± 200 mL of slurry volume. The container should be able to transmit pressures of 50 psi to allow pressure equalisation between the inside and outside of the container.

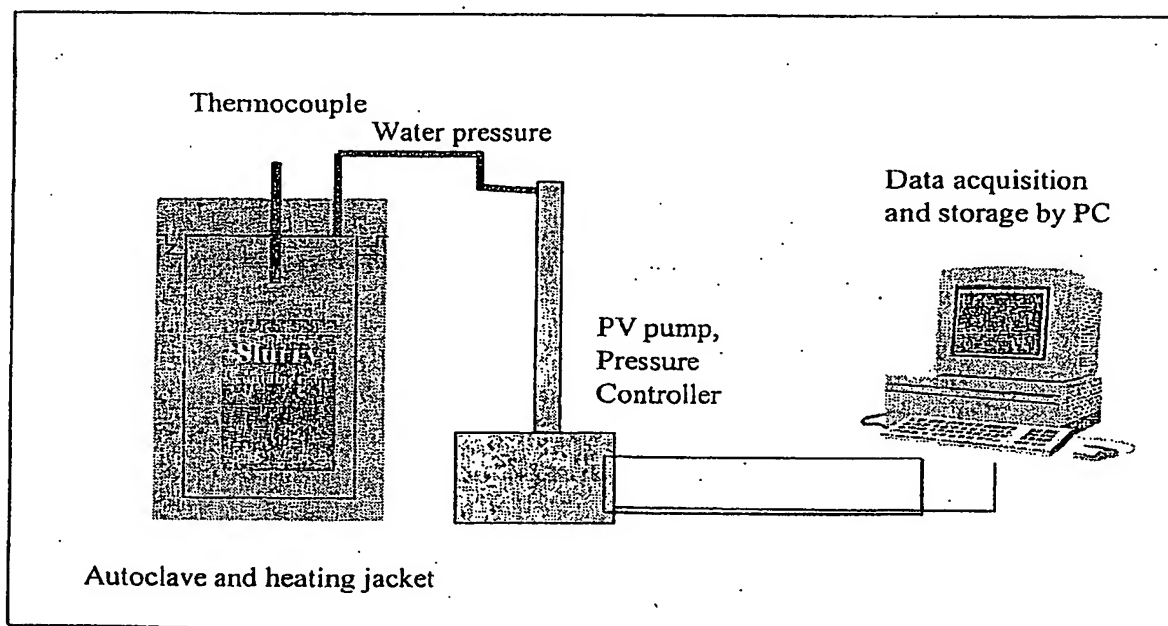


Figure 5

7.2.2 Autoclave

An autoclave suitable for placing the container containing the slurry should be employed. The autoclave shall be equipped with a heater and a temperature controller maintainable within $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$) of the prescribed test temperatures shall be employed. The pressurisation medium is water that is regulated with a pressure/volume (PV) pump. The autoclave is equipped with a thermocouple to measure the temperature inside the cell. The autoclave should have a suitable pressure rating to accommodate tests equivalent to the hydrostatic pressure inside a borehole. The autoclave shall be leak tight for several days, having a leak rate lower than 0.0002 ml/min. The typical volume of an autoclave is 750 to 1500 ml.

7.2.3 Temperature measuring system

The temperature measuring system shall be calibrated to an accuracy of $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$). Calibration shall be no less frequent than monthly. The procedure described in annex A of ISO 10426-2:— is commonly used. Two commonly used temperature measuring systems are:

7.2.3.1 Thermocouple

A thermocouple system with the appropriate range may be used.

7.2.4 Pressurisation pump

A PV pump is required to pressurise the autoclave, which can record the volume pumped and the pressure applied. The pump should be regulated such the a constant pressure ± 10 psi can be maintained for several days, while the time-volume-pressure data are recorded with a data acquisition system interfaced with computer. The volume should be recorded with an accuracy of ± 0.1 ml

7.3 Procedure

7.3.1 Preparation and placement of slurry

7.3.1.1 Slurry

Test samples should be prepared according to clause 5 of ISO 10426-2:—. The mixing device should be calibrated annually to a tolerance of $\pm 3,3$ rev/sec (200 r/min) at 66,7 rev/sec (4000 r/min) speed and $\pm 8,3$ rev/sec (500 r/min) at 200 rev/sec (12 000 r/min).

If larger slurry volumes are needed, an alternate method for slurry preparation is found in Annex A of ISO 10426-2:—. As required, the density of the cement slurry can be determined by methods found in clause 6 of ISO 10426-2:—.

7.3.1.2 Placing slurry in Container

Condition the slurry in the atmospheric consistometer (see 5.2.5) for a period of 20 minutes ± 30 seconds. The bath temperature shall be maintained at $27 \pm 1,7^{\circ}\text{C}$ ($80 \pm 3\text{ }^{\circ}\text{F}$) throughout the stirring period.

The slurry is poured into an impermeable flexible container, which is sealed by leak tight lid. Make sure the lid is absolutely tight so no water from the autoclave can access the slurry during the test. The volume of slurry shall be 500 ± 200 mL.

Settling of slurry must be avoided and the set-time of the slurry should be longer than the heat-up time during the test.

Extreme care is taken so that no air is entrapped in the slurry or at the top of the container. In particular, when one can see an expansion while cement has not yet reached the temperature of the autoclave (beginning of test), it may correspond to thermal expansion of the air and water, which has been trapped. Once container has been prepared it is placed in the autoclave and immersed in water, the autoclave is closed where by all air is removed by displacing the entrapped air with water by an overrun (and pressurisation insert).

The autoclave is connected to the PV pump and pressurised to the test pressure. Then the autoclave is heated to the test temperature. The pressure and volume is recorded throughout the test.

7.3.2 Curing

When the slurry has reached the test temperature the temperature must be held constant for the remainder of the test to avoid volume changes due to temperature expansion or contraction. A typical test may take from one day to three days of hardening.

7.4 Measurement and Calculations

7.4.1 Measurement and Calculations of the expansion or shrinkage of not-compressible slurries

The volume changes after reaching the test temperature are used to calculate the bulk expansion or shrinkage. In this case the volumes caused by heating or compacting air are neglected and the volume after reaching temperature is considered to be the same as the initial volume of the slurry container. The following formula is used to calculate the expansion or shrinkage:

$$\%_{(t)} = 100 \times (V_t - V_i) / V_i$$

(6)

Where:

$$V_t = B_t - B_i$$

V_i = initial volume of the cement slurry, expressed in millilitres (mL)

V_t = final volume of the cement slurry, expressed in millilitres (mL)

B_i = volume of the PV pump after reaching the test temperature, expressed in millilitres (mL)

B_t = volume of the PV pump after time has elapsed, expressed in millilitres (mL)

The bulk expansion or shrinkage shall be plotted as function of elapsed time or reported as 24 hrs, 48 hrs or 72 hrs expansion shrinkage. The calculated bulk expansion or shrinkage shall be reported in relation to the accuracy, eq rounding depending on method's significant figures.

7.4.2 Measurement and Calculations of compressible slurries

To correct for the volume change of compressible slurries and temperature expansion the following correction shall be used:

First determine the volume required (V_p) to pressurise the autoclave containing the sample container filled with water to the test pressure. Then heat the autoclave to the test temperature and determine the volume change

Characterizing Curing-Cement Slurries by Permeability, Tensile Strength, and Shrinkage

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Summary

This work was carried out to obtain more knowledge about the transition period of curing oilwell cements. The results show that the curing characteristics are a function of temperature and that there is a correlation between shrinkage and cement content. The paper also introduces a new mechanism for gas migration and discusses how the studied parameters can be used to predict gas migration.

Introduction

The setting process of cement slurries in oil wells is very complex. Many parameters contribute to the final result, such as geling, shrinkage, temperature, pressure, filter loss, cement structure and strength buildup, slurry permeability, entry pressure, capillary pressure, mud and mud cake, formation properties, well history, and possibly other parameters as well. Some of these parameters are next to impossible to characterize. Others are simple to measure in a laboratory setup, but may not reflect downhole conditions. The cement setting process has been investigated extensively, but there are still many factors not fully understood. We have tried to extend the knowledge by monitoring temperature evolution, hydrostatic pressure, permeability, tensile strength, and total chemical shrinkage during hydration. We will in turn discuss the importance of these parameters.

Cement hydration is an exothermic reaction which can be observed as a temperature increase. Temperature is easy to measure, and the shape and peak of the temperature curve give valuable information on the hydration process, i.e., hydration onset and rate. The hydrostatic pressure is important as gas flow into the cement will be initiated when the pressure of the cement column falls below that of a gas bearing formation.¹⁻³ This pressure drop is due to cement shrinkage at the same time as the shear strength develops, enabling the cement to hang onto the wellbore and casing.

But what mechanism will govern inflow of gas when the cement pressure has dropped far enough? We think the capillary entry pressure of the cement pore structure is important. When exposing gas to the water-saturated cement, the nonwetting gas phase has to overcome the entry pressure of the cement pore system due to the interfacial tension between cement pore fluid and gas. The entry pressure is high when the pores are small while the permeability on the other hand is low, and entry pressure is, in general, inversely proportional to permeability. After having overcome the entry pressure, the relative permeability and the differential pressure between the formation gas and the cement column

control how much cement pore water will be displaced by gas. This is a complicated process where the permeability and pressure change continuously.

Considering how important the permeability is for governing flow into and through the cement pores, surprisingly little work has been carried out on permeability during setting. Sutton and Ravi⁴ state that low fluid loss slurries exhibit a permeability of less than 100 md at a static gel strength of 200 lb/100 ft² and that it approaches 5 md at 500 lb/100 ft². Plee *et al.*⁵ have studied the permeability of bentonite-cement slurries and found a typical value of 50 to 100 md for fresh slurries and that decreasing permeability correlates linearly with increasing surface area. Only Appleby and Wilson⁶ have monitored the permeability at several points of time up till and past final set. Their results show an initial permeability about 1 darcy, falling down to around 1 md at the temperature peak.

When the cement slurry is in the process of losing its hydraulic (liquid) properties, the strength of the cement matrix is still low. The pressure difference between the formation gas and the hydrostatic pressure of the cement slurry may overcome the strength of the matrix. In this situation it is the compressional strength of a confined cement which is of importance and this strength parameter is the highest one. Therefore, we think that it is not very probable that gas will break the matrix from outside and migrate through the created microfractures. However, if gas has entered the cement through its pores, it is the tensile strength of the cement which has to be overcome in order to break the cement matrix. The tensile strength is the lowest strength parameter, and we believe that this is a likely mechanism of fracturing the cement, leading to gas migration.

We, therefore, propose a new mechanism for initiating gas migration. After the gas has entered the pore system of the cement, the gas inside may overcome the tensile strength of the cement structure, break the cement matrix, and migrate through the microfractures. During the hydration process all the studied parameters change continuously. The hydrostatic pressure inside the cement slurry column drops and the gas bubbles already inside the cement will, therefore, try to expand. The decreasing pore radii of the cement matrix stop this expansion and the bubbles may maintain their pressure while the cement pressure will continue to decrease. This leads to a pressure difference which may be large enough to overcome the tensile strength of the cement structure and thus creating fractures.

The hydrostatic pressure drop of the cement column is mainly governed by the chemical shrinkage of the cement. Before initial set, a low shrinkage rate is preferable because the resulting hydrostatic pressure decline will be slower than for a slurry with a higher shrinkage rate. Slow shrinkage has two advantages: pressure equilibrium between formation and slurry column is reached at a later point of time and afterwards the pressure difference between formation and slurry, the driving force behind flow of pore fluid into the cement, will be lower. Both factors should reduce the risk of early time gas migration. After initial set, how-

*Now with the Geological Survey of Norway.

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This paper (SPE 57712) was revised for publication from paper SPE 38267, first presented at the 1997 SPE Western Regional Meeting held in Long Beach, California, 25-27 June. Original manuscript received for review 25 June 1997; Revised manuscript received 13 May 1999. Paper peer approved 21 June 1999.

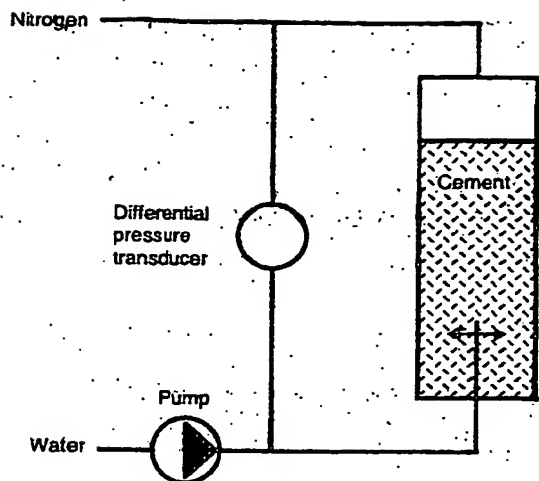


Fig. 1—Principle of the permeability and fracturing setup: water is pumped into the cement causing a pressure increase.

ever, when high shrinkage and pressure loss are inevitable, a short transition time will reduce the time during which gas migration can be initiated.

The chemical shrinkage may be divided in two parts, external and internal. The external shrinkage is the bulk or external dimensional volume change of the slurry leading to a possible microannulus between the cement and the wellbore. The internal chemical shrinkage is caused by formation of contraction pores which contribute to the connectivity between pores in a set cement, and hence, to permeability. The total chemical shrinkage is the sum of the external and internal shrinkage.

Chemical shrinkage of oilwell cements has been investigated by several researchers. The most extensive tests have been carried out by Chenevert and Shrestha,⁷ and by Sabins and Sutton,⁸ but other papers have also been presented.⁹⁻¹¹ The applied temperatures and pressures range from ambient conditions up to 224 °C and 121 MPa.⁷ Both total⁸⁻¹¹ and external chemical shrinkage^{7,9} were reported with a total shrinkage at 20 or 24 hours varying from 0.6 to 6 vol%, while most results were in the range of 1.5 to 3 vol%. Only Sabins and Sutton⁸ have performed measurements of external shrinkage under realistic downhole conditions. According to them, most of this shrinkage occurs when the slurry still is plastic. Their results showed an average of 0.16 vol% and from this they calculated the contraction pores to account for 97.5 to 99% of the total shrinkage. Thus, from a gas migration point of view, the formation of contraction pores is by far the largest and most important part of the chemical shrinkage.

Experimental Setup and Procedure

Permeability and Tensile Strength. The permeability and the tensile strength of a cement slurry were measured in the same cell where measurements can be done at temperatures up to 200 °C and at pressures up to 20 bars. In addition, the temperature evolution and hydrostatic pressure were recorded. Fig. 1 shows the setup with the cell filled with cement slurry where the diameter of the cell is 5 cm and the height of the cement column is 25 cm. The water inlet is placed 5 cm from the bottom of the cell. By using four cells, measurements can be done at four different time points after mixing, and thus, generating a trend curve. To map the whole permeability and strength development 2 to 4 of these four-cell measurements had to be carried out. The top of the cell, above the cement slurry, is filled with nitrogen gas, and the system is pressurized from 15 to 20 bar. A pressure transducer measures the

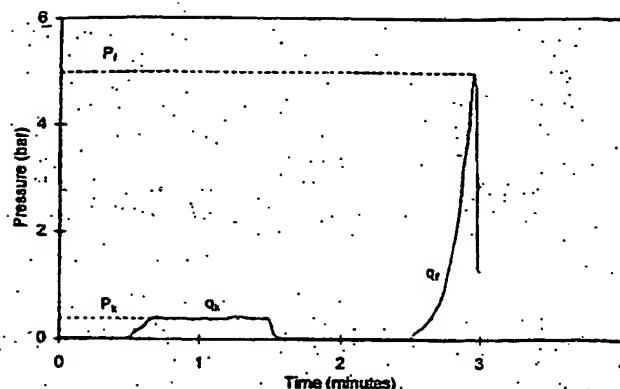


Fig. 2—Pressure behavior during a permeability and strength test.

differential pressure between the water and the gas lines which is used to find both the permeability and tensile strength. All parameters were recorded by a data acquisition card and stored in a personal computer. The slurries were prepared and mixed in accordance with API Spec. 10.¹²

Before a new slurry was tested for permeability and strength, one nondestructive test was carried out to obtain complete temperature and pressure curves for the whole setting process. This initial run was also an aid to choose the points of time when to run the permeability and strength tests.

To find the permeability, a small rate of water ($q_1 = 0.1$ to 0.33 mL/min) was pumped into the cement slurry until a constant pressure p_1 was obtained. This is shown in Fig. 2 where the first part of the pressure/time curve represents this part of the experiment. When the pressure reached a constant level during pumping, the flow was assumed to be Darcy flow, enabling computation of the slurry permeability.

The tensile strength measurements are based on the principle of hydraulic fracturing and were usually done 1 minute after the permeability test. A high rate of water ($q_2 = 5$ to 20 mL/min) was pumped into the cement slurry and the pressure increased rapidly to a maximum value p_2 . At this point the tensile strength of the cement slurry was exceeded, a fracture developed, and the pres-

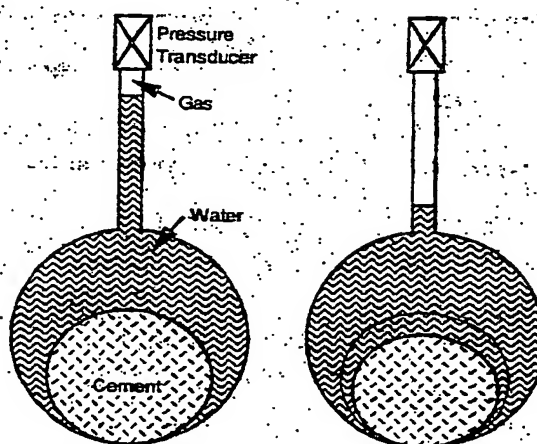


Fig. 3—Principle of the shrinkage measurements: initial conditions in the left-hand figure and to the right, conditions after some time.

TABLE 1—CEMENT SLURRY DATA, RECIPE AND RESULTS

		Slurry							
		T	B	A	C	D	E	F	G
		Slurry Data							
Test Temperature, °C		90	180	140	140	140	140	140	140
Slurry Density, g/cm ³		1.90	2.15	2.05	2.03	2.06	2.07	1.98	1.88
		Slurry Recipe							
Additive	g/cm ³								
API Class G Cement	3.22	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Fresh Water, l/hk*	1.00	43.24	54.86	45.52	39.96	44.28	44.28	44.28	44.28
Weight Material, % bwoc**	4.85		74.87	38.77	13.88	13.88	13.88	13.88	
Antistrength Retrogr., % bwoc	2.65		26.03	25.97	30.00	30.00			
Antigas Migration, l/hk	1.40		13.00	13.00	12.00				
Dispersant, l/hk	1.21		3.00	3.00	3.00	3.00	3.00	3.00	3.00
Retarder, Low Temp., l/hk	1.20	0.80							
Retarder, Medium Temp., l/hk	1.18			1.25	1.25	1.25	1.25	1.25	1.25
Retarder, High Temp., % bwoc	1.25		0.80						
Antifluid Loss, l/hk	1.04		3.00	5.00	3.00				
Calcite Flour % bwoc	2.72						30.00		
		Gas Tightness							
Gas Tightness in Test Rig		Leak	Tight	Tight	—	Tight	—	—	—
Gas Tightness Number, F_{gt}		21.20	4.9	0.6	—	3.5	3.9	4.7	8.0
		Shrinkage							
Cement, vol%		41.35	23.74	26.63	29.73	33.12	33.22	37.67	39.02
Shrinkage at 20 hours, vol%		3.92	2.61	1.79	1.98	2.58	2.49	2.84	3.11
Shrinkage at 20 hours, mL/100 g Cement		2.94	3.41	2.09	2.06	2.42	2.33	2.34	2.48

*Liters per hundred kilos of cement.

**Percent by weight of cement.

sure dropped instantly. In Fig. 2 the second peak of the pressure/time curve represents the fracturing pressure. After the test, a horizontal fracture at the level of the water inlet could be observed in the cement and the maximum strength that can be measured with the apparatus is 5 bar. At this strength the cement has reached final set and is quite hard (for comparison, sandstones have a tensile strength of 40 to 150 bar).

Shrinkage. For the shrinkage measurements a second cell was made. The measurements were based on the pressure/volume relationship of a gas. The principle is shown in Fig. 3, indicating initial conditions in the left-hand figure. At a later stage, shown in the right of Fig. 3, the shrinkage process leads to a lower gas pressure. When the initial temperature, pressure, and volumes are known, the shrinkage can be calculated on the basis of the continuously declining pressure during the test. The calculation is also corrected for gas temperature. Total shrinkage was measured by placing the cement in a slightly permeable paper cup containing around 50 mL of slurry. All samples were weighed before and after each test, and the volume of the hardened sample was found by applying Archimedes' principle. The applied pressure was in the range of 6 to 16 bar, which was measured with an accuracy of $\pm 0.15\%$. All parameters were recorded by a data logger and stored in a personal computer.

The method works well but has some drawbacks. It requires precision in the initial values and it is sensitive to temperature changes. The shrinkage calculations are taken from the point of maximum pressure or when the test temperature stabilizes because we were unable to quantify the water expansion that occurs before

this point of time. This has the advantage that any shrinkage occurring before placement of the cement in a well will not be taken into account, even though some initial shrinkage may not be recorded.

Results and Discussion

Our tests were mainly performed at 140 °C, although the test program also included experiments at lower and higher temperatures as well. The recipes and data of the slurries presented in this paper are shown in Table 1. Slurry T is a very simple test mixture which

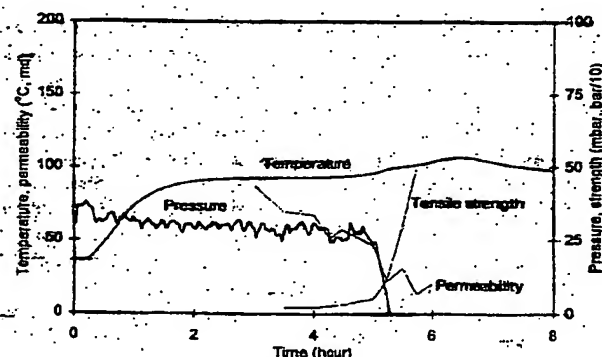


Fig. 4—Slurry T at 90 °C: temperature, pressure, strength, and permeability.

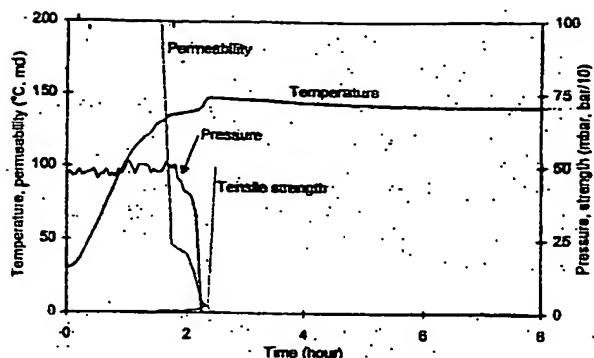


Fig. 5—Slurry A at 140 °C: temperature, pressure, strength, and permeability.

is not gas tight, while slurries A and B are commercial recipes containing all the necessary additives to avoid gas migration. Slurries C-G will be discussed below. The permeability, tensile strength, and shrinkage results of slurries T, A, and B are presented in Figs. 4 through 9, where the shrinkage is given as both vol% and mL/100 g cement. The mL/100 g values are a measure of degree of hydration.

It is difficult to compare our permeability results with some of those from the literature^{4,5} because of differing conditions. However, Appleby and Wilson's⁶ data compare well with our results where the curve shapes and the permeabilities at the temperature peak are similar. Our shrinkage results at 20 hours fall within the range presented in the literature.⁷⁻¹¹

Temperature Influence. From Figs. 4 through 9, it is evident that there is a considerable difference in behavior between the three slurries at 90, 140, and 180 °C. For slurry A at 140 °C all parameters change very rapidly when the hydration starts, whereas the other two slurries at 90 and 180 °C are slower. For all three slurries the temperature peaks coincide with the most marked changes of the other measured parameters, and the higher the cement temperature is above the set temperature, the more rapid are these changes. As an example, slurry A with the most pronounced temperature peak also shows the fastest initial shrinkage up to 1 vol% and thereafter a decaying shrinkage rate and temperature. The temperature peak of slurry B is the lowest corresponding to the slowest initial shrinkage (see Fig. 9). The same is true for its permeability and tensile strength. This correlation is to be expected as the temperature evolution is a measure of the rate of

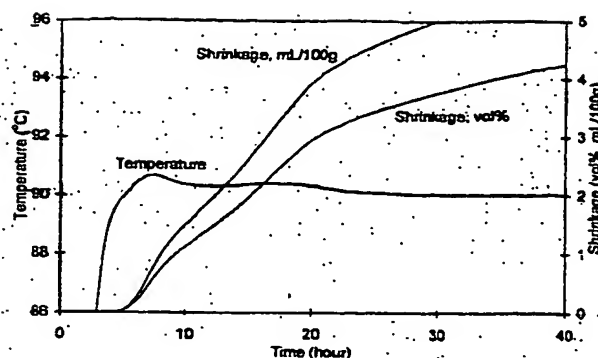


Fig. 7—Slurry T at 90 °C: temperature and shrinkage.

hydration, and hence, the parameter behavior. Later in the curing process, after around 15 hours, the shrinkage measured in mL/100 g is higher for slurries T and B than for slurry A. More interesting from a hydration point of view, though, are the two temperature peaks of slurries T and B, the first being at 5 to 7 hours and the second around 15 to 20 hours, seen in Figs. 7 and 9.

We also tested several other slurries at the temperatures 90, 140 and 180 °C and they exhibited the same behavior, demonstrating that this is a general trend. A closer look at the literature also confirm this behavior. The freshwater-based slurries of Chenevert and Shrestha⁷ at 93, 121, and 177 °C are not strictly comparable as they measured the external and not total shrinkage. Nevertheless, their medium temperature slurry does show the fastest initial shrinkage. Sabins *et al.*¹³ carried out a substantial number of compressive strength tests and their 24-hour results are plotted in Fig. 10. Apart from two slurries at 143 °C with a strength above 20 MPa, the strength of the cements above 135 °C is lower than those below this temperature. Sabins and Samon¹⁴ extended this work and the results follow the same trend although not so clearly.

However, one question remains, how can this temperature influence be explained? The recipes of the slurries A and B at 140 and 180 °C, respectively, are very similar, but their behavior is quite different, implying that this difference may be a temperature effect on the cement hydration chemistry. We leave this question open.

Gas Tightness. With respect to gas migration, a short transition period (as with slurry A) will limit the period in which gas can enter the cement, thus reducing the hazard of gas migration. All three slurries were also tested in the gas migration rig of Jamali *et al.*¹⁵ where the T slurry was leaking and the other two were

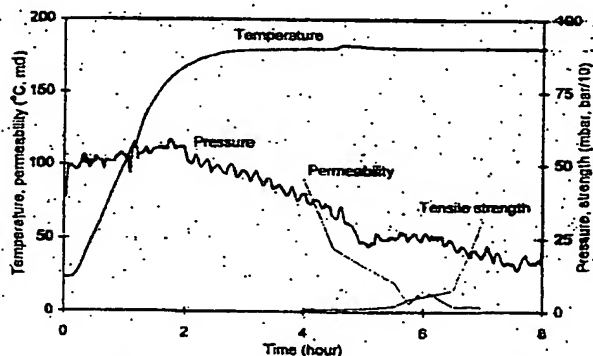


Fig. 6—Slurry B at 180 °C: temperature, pressure, strength, and permeability.

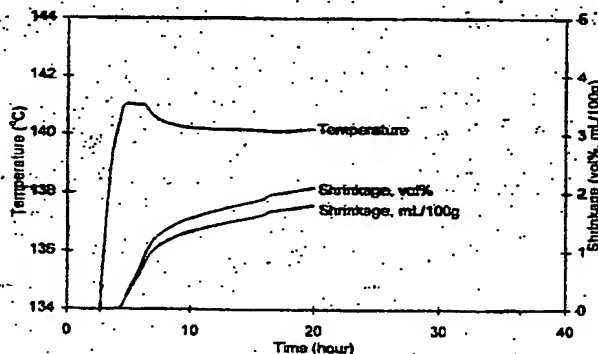


Fig. 8—Slurry A at 140 °C: temperature and shrinkage.

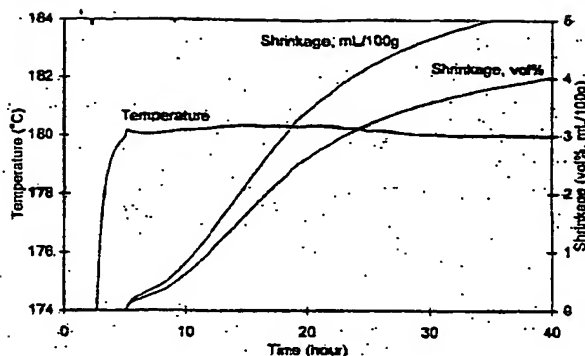


Fig. 9—Slurry B at 180 °C: temperature and shrinkage.

tight. This may be due to the simple composition of this slurry.

In order to relate the hydrostatic pressure, tensile strength, and permeability to gas tightness, a systematic interpretation of the results for several slurries were performed. For the untight cements, the hydrostatic pressure dropped almost immediately down to the water column pressure (30 mbar in Fig. 4), stayed constant for some time, and then fell abruptly. The gas tight pastes in Figs. 5 and 6 were better at maintaining the hydrostatic cement pressure until the onset of hydration. Buildup of tensile strength was generally also faster for the tight cements, indicating the importance of a short transition period.

Based on the tensile strength buildup we have defined a time window, $\Delta t = t_2 - t_1$. The two points of time t_1 and t_2 represent a tensile strength of 0.3 and 5 bars, respectively. At time t_2 the cement is hard. The strength buildup should be rapid, thus Δt should be small. For the basic slurry, we found in Fig. 4 that Δt is 2 hours, while in Fig. 5 Δt is 15 minutes for the gas tight slurry A. Slurry B at 180 °C exhibited a time window of 1.6 hours. At the same points of time, the permeabilities k_1 and k_2 are recorded. In order to avoid gas intrusion into the pores, the permeability should be low within the time window. The theoretical initial hydrostatic pressure of the fresh cement slurry is denoted p_i . The drop of the hydrostatic pressure at the time when the strength starts building up, i.e., p_1 at t_1 , should be small. Therefore, the value of $(p_i - p_1)$ should be small.

The three parameters, the time window, the permeabilities, and the hydrostatic pressures may be combined in various ways to find

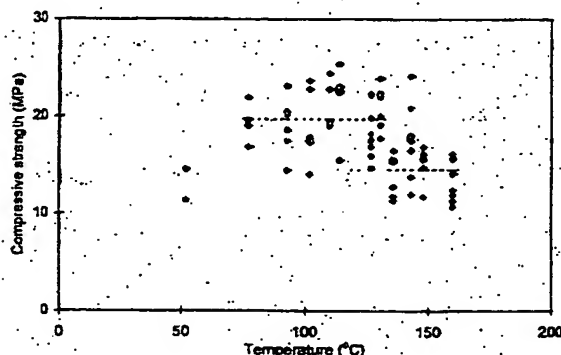


Fig. 10—Compressive strength at 24 hours. The dotted lines represent the average within the two temperature ranges (excluding the two gray points). Data from Sabins et al. (Ref. 13).

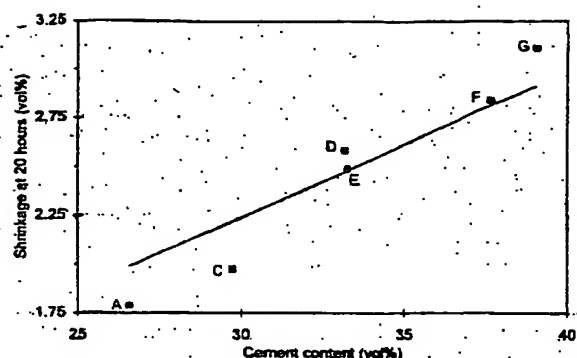


Fig. 11—Shrinkage at 20 hours vs. cement content.

a factor that can characterize a cement's ability to resist gas migration. We have tested the following combination of the parameters, called the gas tightness factor:

$$F_{gt} = \sqrt{\Delta t} \times \frac{k_1 - k_2}{k_1} \times k_2 \times \left(\frac{p_i - p_1}{p_i} + 1 \right) \quad (1)$$

From the discussion above, we conclude that the factor should be as small as possible to have a gas-tight cement.

In Table 1 the leaking slurry T has a F_{gt} value above 20 whereas the three tight slurries exhibit values below 5. Thus, based on these slurries, the threshold is somewhere between 5 and 20. To further test the F_{gt} factor, slurry A was used as a starting point for simplifying the cement composition to study the influence of additives on gas migration. The recipes and F_{gt} factors of slurries D-G are shown in Table 1 and the results show that simplifying the recipe will increase the F_{gt} factor. These slurries were not tested in the gas rig, but slurry G with a value of 8 would probably leak due to its simple recipe. Later results, however, show some overlap between tight and leaking slurries, but the general trend is still clear, i.e., a short transition period is essential for a gas-tight cement.

Shrinkage vs. Cement Content. With one addition, the 140 °C slurries tested for the F_{gt} factor were also tested for shrinkage. The recipes and data of slurries C-G are shown in Table 1. The shrinkage at 20 hours was found to be correlated to the cement content, as shown in Fig. 11, where the curve is forced through zero, as zero-cement content should yield no shrinkage. This correlation is self-evident and has been reported previously^{7,8} without being linked to the gas migration problem, however. As long as the strength development of the cement slurry is satisfactory, more use of inert extender will reduce the shrinkage and the risk of gas migration. The data in Table 1 support this conclusion, where the only leaking recipe, slurry T, shows the highest absolute shrinkage at 20 hours.

Conclusions

1. Two cells were successfully developed to study hydrostatic pressure, temperature evolution, tensile strength, permeability, and total shrinkage during cement hydration.

2. A new mechanism for gas migration is proposed. First, the gas has to overcome the entry pressure of the cement pores, and once inside the cement (caused by the continuously changing pressure conditions) the gas may fracture the cement structure.

3. There is a marked contrast in behavior of slurries at 90, 140 and 180 °C where the 90 and 180 °C slurries exhibit two temperature peaks. This difference is most likely due to a temperature effect on the cement hydration chemistry.

4. To predict migration problems, a gas-tightness factor based on the collected data was defined and compared to results in a gas migration test rig. A short transition period is important.

5. There is a positive correlation between total chemical shrinkage and cement content. A low shrinkage will reduce the risk of gas migration.

Nomenclature

- F_{gt} = gas tightness factor as defined in Eq. (1)
 k = permeability, L^2 , md
 p = pressure, m/L^2 , bar, mbar, MPa
 q = flow rate, L^3/t , mL/min
 t = time, t , hour, minute
 Δt = time difference, t , hour, minute

Subscripts

- 1,2 = point of time
 f = fracture
 i = initial
 k = permeability

Acknowledgments

The authors would like to extend their thanks to the sponsors of this work: The Research Council of Norway, Norsk Hydro A/S, Saga Petroleum A/S, and Statoil A/S, and their respective representatives Svein Joakimsen, Øystein Kälvenes, Jan-Erik Ølvin, and Nils-Egil Nøddland for their personal engagement and contributions. We also thank Vincent H. J. Bosch for carrying out some of the experiments, and Harald Justnes and John Jamth for valuable discussions.

References

- Carter, G. and Slagle, K.: "A Study of Completion Practices to Minimize Gas Communications," *JPT* (September 1972) 24, 1170.
- Tinsley, J.M. et al.: "Study of Factors Causing Annular Gas Flow Following Primary Cementing," *JPT* (August 1980) 32(8): 1427.
- Cooke, E., Kluck, M.P., and Medrano, R.: "Field Measurements of Annular Pressure and Temperature During Primary Cementing," *JPT* (August 1983) 35(9): 1429.
- Sutton, D.L. and Ravi, K.M.: "New method for Determining Downhole Properties That Affect Gas Migration and Annular Sealing," paper SPE 19520 presented at the 1989 SPE Annual Technical Conference and Exhibition, San Antonio, 8-11 October.
- Plec, D. et al.: "Microstructure, Permeability and Rheology of Bentonite-Cement Slurries," *Cem. Concr. Res.* (January 1990) 20(1): 45.
- Appleby, S. and Wilson, A.: "Permeability and Suction in Setting Cement," *Chem. Eng. Sci.* (January 1996) 51(2): 251.
- Chenevert, M.E. and Shrestha, B.K.: "Chemical Shrinkage Properties of Oilfield Cements," *SPEDE* (March 1991) 6(1): 37.
- Sabins, F.L. and Sutton, D.L.: "Interrelationship Between Critical Cement Properties and Volume Changes During Cement Setting," *SPEDE* (June 1991) 6(2): 88; *Trans., AIME*, 291.
- Justnes, H. et al.: "Chemical Shrinkage of Oil Well Cement Slurries," *Adv. Cem. Res.* (April 1995) 7(26): 85.

- Parcevaux, P.A. and Sault, P.H.: "Cement Shrinkage and Elasticity: A New Approach for a Good Zonal Isolation," paper SPE 13176 presented at the 1984 SPE 59th Annual Technical Conference and Exhibition, Houston, 16-19 September.
- Stewart, R.B. and Schoum, F.C.: "Gas Invasion and Migration in Cemented Annuli: Causes and Cures," *SPEDE* (March 1988) 3(1): 77.
- Spec. 10, *Specification for Materials and Testing for Well Cements*, fifth ed., API, Dallas (1990).
- Sabins, F.L., Sutton, D.L., and Cook, Jr., C.: "Effect of Excessive Retardation on the Physical Properties of Cement Slurries," *JPT* (August 1984) 36(9): 1357.
- Sabins, F.L. and Sutton, D.L.: "The Relationship of Thickening Time, Gel Strength and Compressive Strength of Oil Well Cements," *SPEDE* (March 1986) 1(2): 143.
- Jamth, J. et al.: "Large scale testing system to evaluate the resistance of cement slurries to gas migration during hydration," paper 95-405 presented at the 1995 CADE/CAODC Spring Drilling Conference, Calgary, 19-21 April.

SI Metric Conversion Factors

bar	X	1.0*	E-01	=	MPa
°F	(°F - 32)/1.8			=	°C
ft	X	3.048*	E-01	=	m
g/cm ³	X	1.0*	E+03	=	kg/m ³
in.	X	2.54*	E+00	=	cm
lb/100 ft ²	X	4.788 026	E-01	=	Pa
lbm	X	4.535 924	E-01	=	kg
md	X	9.869 233	E-04	=	μm ²
mL	X	1.0*	E+00	=	cm ³
psi	X	6.894 757	E-03	=	MPa
gal	X	3.785 412	E-03	=	m ³

*Conversion factors are exact.

SPEDE

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ISO/ TC 67/SC3

Date: 20/05/02

ISO/WD 10426-5 (v1.5) : (E)

ISO/ TC 67/SC3/ WG2

Secretariat: NTS

Petroleum and natural gas industries - Cements and materials for well cementing – Part 5: Test methods for determination of shrinkage and expansion of well cement formulations

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Document type : International standard

Document subtype : Not applicable

Document stage : (20) Preparatory

Document language : E

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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International Standard ISO 10426-5 was prepared by Technical Committee ISO/TC 67 *"Materials, equipment and offshore structures for petroleum and natural gas industries, Sub-Committee SC3 Drilling and completion fluids, and well cements"*.

ISO 10426 consists of the following parts, under the general title *Petroleum and natural gas industries - Cements and materials for well cementing*:

Part 1: Specification.

Part 2: Testing of well cements.

Part 3: Testing of deepwater well cement formulations

Part 4: Methods for atmospheric foamed cement slurry preparation and testing

Introduction

Users of this part of ISO 10426 should be aware that further or differing requirements may be needed for individual applications. This part of ISO 10426 is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This may be particularly applicable where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this International Standard and provide details.

Dimensional change after placement in oil and gas well cement slurry, a phenomenon often referred to as shrinkage, has often been used to explain various problems (e.g. microannulus leading to bad cement bond logs, interzonal communication leading to costly remedial jobs and also lack of a seal of cement inflatable packers). Attempts have been made to find additives to decrease shrinkage, but shrinkage (as well as the exothermal reaction) is closely related to the hydration of the cement. The best solution for this shrinkage so far has been the identification of additives that favor the expansion of the cement slurry. However, even if cement expands dimensionally, it still will shrink internally. In this case, the bulk expansion of the cement sample is simply superposed on an inner shrinkage that will affect the porosity of the sample. Shrinkage is not just one property of cement, such as rheology or thickening time that can be defined by one measurement.

Shrinkage and expansion in cement slurry result from the formation of hydration products having a different density from the compounded density of the reaction components. This can result in:

- Change in pore volume
- Change in pore pressure
- Change in sample dimensions
- Change in internal stress.

The change in sample dimensions will be referred to as bulk shrinkage or bulk expansion. The change in pore volume will be referred to as chemical inner (hydration) shrinkage. Shrinkage and expansion of cement slurry refer to the result of the measurement of a volume change in cement slurry. This will be expressed in percent by volume. The volume to which all volume changes are related, is the volume of the slurry immediately after mixing and placement in the experimental equipment.

In this part of ISO 10426, units are given as SI, and where practical, U.S. Customary units are included in brackets for information.

Petroleum and natural gas industries - Cements and materials for well cementing – Part 5: Test methods for determination of shrinkage and expansion of well cement formulations

1 Scope

This part of ISO 10426 provides the methods for the testing of well cement formulations determine the volumetric changes during the curing process.

2 Normative reference

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10426:2000, Petroleum and natural gas industries - Cements and materials for well cementing: Part 1 Specification

ISO 10426:—¹⁾ Petroleum and natural gas industries - Cements and materials for well cementing: Part 2 Recommended practice for testing of well cements.

3 Terms and definitions

For the purposes of this part of ISO 10426, the following terms and definitions apply.

3.1

Bulk expansion

an increase in the external volume (dimensions) of a cement sample

3.2

Bulk shrinkage

a decrease in the external volume (dimensions) of a cement sample

3.3

Inner expansion

the volumetric change of a cement sample measured by the amount of fluid expelled from the cement

3.4

Inner shrinkage

the volumetric change of a cement sample measured by the amount of fluid entering the cement

¹⁾ To be published

3.5

Chemical or hydration shrinkage

the reduction of volume of product hydrates versus the volume of anhydrous compounds plus water (under fixed boundary conditions inner shrinkage = chemical shrinkage).

4 Sampling

4.1 General

Samples of the neat cement or cement blend, solid and liquid additives, and mixing water are required to test a slurry according to this Test Methods. Accordingly, the best available sampling technology should be employed to ensure the laboratory test conditions and materials match as closely as possible those found at the wellsite. Some commonly used sampling devices may be found in Figure 1 of ISO 10426-2:—.

4.2 Method

Applicable sampling techniques for the fluids and materials used may be found in clause 4 of ISO 10426-2:—.

5 Determination of linear shrinkage or expansion under conditions of free access of water: annular ring test

5.1 General information

The annular mould is a device suitable to measure the bulk linear shrinkage or expansion properties of cement composition. The magnitude of expansion depends on the amount of expanding agent, cement powder, slurry design and curing condition (pressure, temperature, time, fluid access). Besides, it should be noticed that expansion is a strong function of boundary condition. The chemical process of mineral growth is strongly controlled by the state of stress, and mineral growth will tend to occur where the stress value is the lowest, i. e. in pore space or empty spaces. Therefore, the cement shrinkage/expansion is not just one material property, which could be uniquely defined. The test does not represent fully the annulus of a well.

5.2 Apparatus

5.2.1 Mould

Use corrosion-resistant metal (i.e. stainless steel). The O.D. of inner ring shall be 50,8 mm (2 inches) and I.D. of outer expansion ring shall be 88,9 mm (3,5 inches). Figure 1,2 and 3.

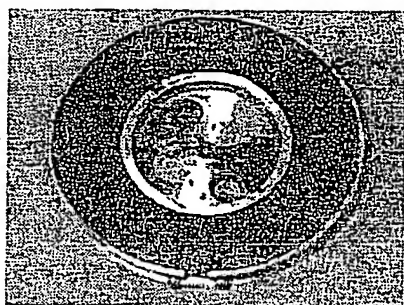


Figure 1

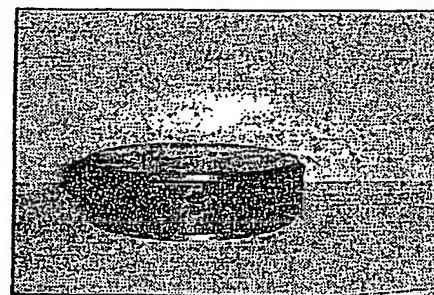
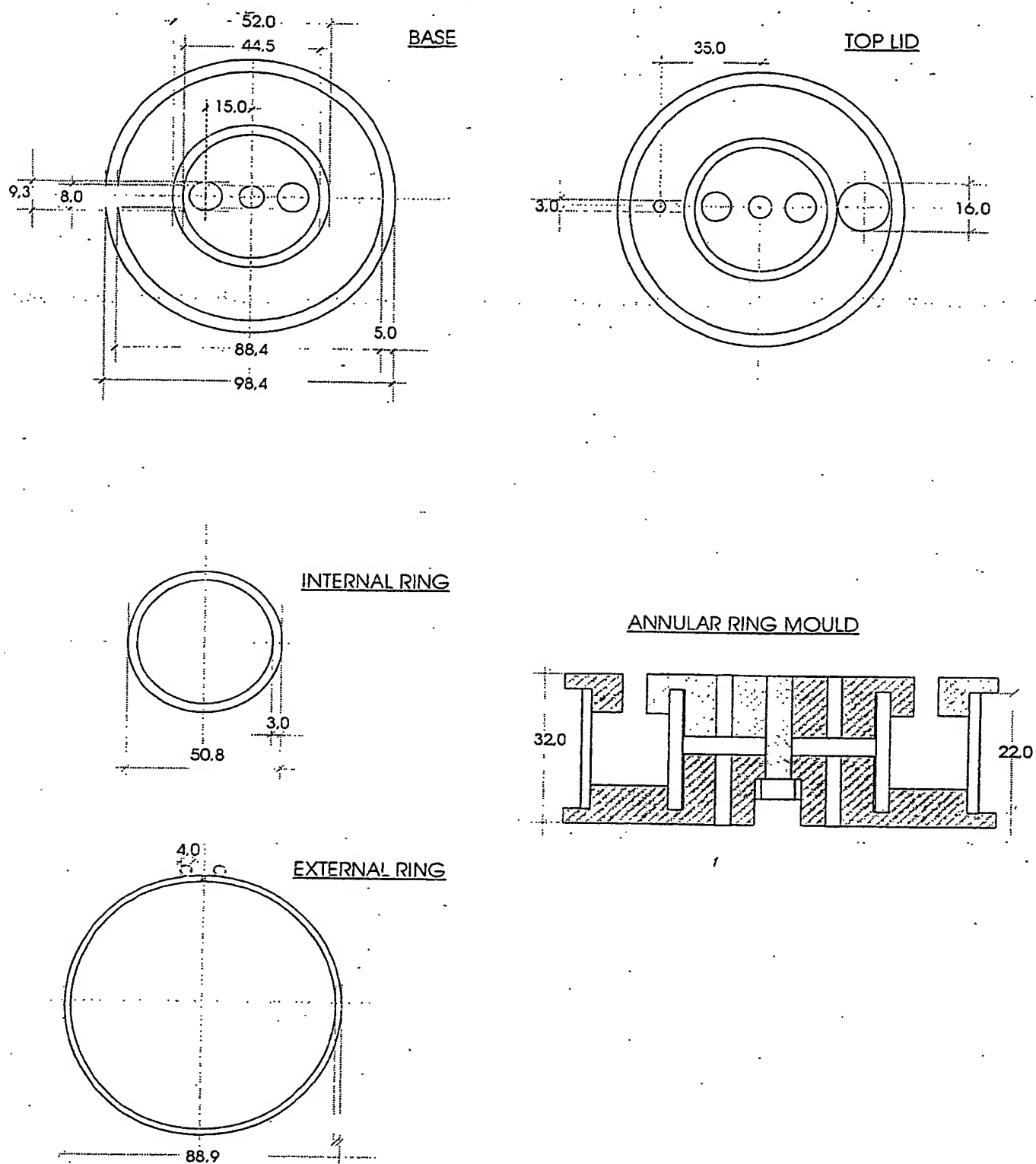


Figure 2



All measures are expressed in mm.

Figure 3

5.2.1.1 Ring calibration

The resilience of the ring of mould shall be calibrated annually. The resilience shall be such that the action mass of $1000 \pm 1\text{ g}$ applied as shown in figure 4 shall increase the distance between the two indicator balls of $2 \pm 0,3\text{ mm}$ ($0,0787 \pm 0,0118$ inches) without permanent deformation.

Attention must be paid to ensure that the load applied is perpendicular to the gap (90°) in order to avoid error, which might be easily made. The readings must be repeated at least three fold to obtain an average value with St. Dev. 0,05.

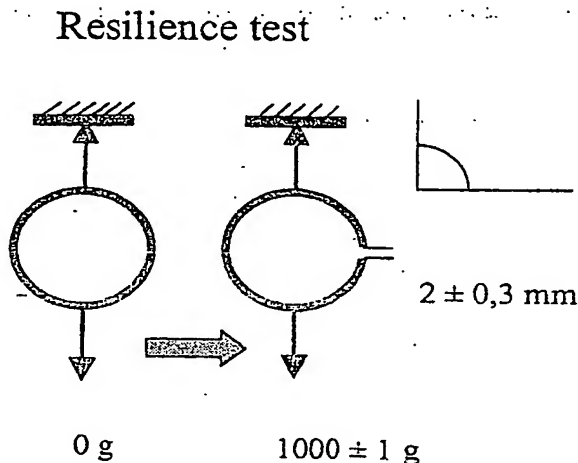


Figure 4

5.2.1.2 Spacer block

The spacer block shall be used only in case of shrinkage measurement. The dimension of block should be 3,175 to 6,35 mm square ($0,125$ to $0,25$ inches) and 22,225 mm tall ($0,875$ inch). To ensure that the spacer block's thermal expansion properties are the same as that of the expandable outer ring, the block should be made of stainless steel.

5.2.2 Water curing bath

A curing bath or tank having dimensions suitable for the complete immersion of a mould(s) in water and capable of being maintained within $\pm 2\text{ }^\circ\text{C}$ ($\pm 3\text{ }^\circ\text{F}$) of the prescribed test temperatures shall be employed. The two types of water curing baths are:

5.2.2.1 Atmospheric pressure apparatus (bath)

An atmospheric pressure apparatus (bath) for curing specimens at temperatures of $90\text{ }^\circ\text{C}$ ($194\text{ }^\circ\text{F}$) or less shall have an agitator or circulating system.

5.2.2.2 Pressurised apparatus

A pressurised apparatus suitable for curing specimens at the appropriate final test temperature $\pm 2\text{ }^\circ\text{C}$ ($\pm 3\text{ }^\circ\text{F}$), and at pressure that can be controlled at least $20,7\text{ MPa} \pm 0,345\text{ MPa}$ ($\pm 50\text{ psi}$) shall be used. The vessel

shall be capable of fulfilling the appropriate schedules described in clause 7 of ISO 10426-2:—, or a schedule designed to simulate a specific well's conditions.

5.2.3 Cooling bath

The cooling bath dimensions shall be such that the specimen to be cooled from the curing temperature can be completely submerged in water maintained at $27\text{ °C} \pm 3\text{ °C}$ ($80\text{ °F} \pm 5\text{ °F}$).

5.2.4 Temperature measuring system

The temperature measuring system shall be calibrated to an accuracy of $\pm 2\text{ °C}$ ($\pm 3\text{ °F}$). Calibration shall be no less frequent than monthly. The procedure described in annex A of ISO 10426-2:— is commonly used. Two commonly used temperature measuring systems are:

5.2.4.1 Thermometer

A thermometer with a range including 21 °C to 100 °C (70 °F to 212 °F) with minimum scale divisions not exceeding 1 °C (2 °F) may be used.

5.2.4.2 Thermocouple

A thermocouple system with the appropriate range may be used.

5.2.5 Consistometer

The atmospheric pressure consistometer shall be used for stirring and conditioning the cement slurry. The consistometer consists of a rotating cylindrical slurry container, equipped with an essentially stationary paddle assembly, in a temperature controlled liquid bath. The consistometer shall be capable of maintaining the test temperature of the bath at $\pm 1,7\text{ °C}$ ($\pm 3\text{ °F}$) and of rotating the slurry container at a speed of $2,5\text{ rev/sec}$, $\pm 0,25\text{ rev/sec}$ (150 r/min , $\pm 15\text{ r/min}$) during the stirring and conditioning period for the slurry. The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials, in accordance with ISO/DIS 10426-1:2000.

5.3 Procedure

5.3.1 Preparation of moulds

The assembled moulds shall be watertight. The interior faces of the moulds and the contact surfaces of the plates are commonly lightly coated with release agent, but may be clean and dry. Place spacer block inside the split of outer ring only in case of shrinkage test measurement.

5.3.2 Preparation and placement of slurry

5.3.2.1 Slurry

Test samples should be prepared according to clause 5 of ISO 10426-2:—. The mixing device should be calibrated annually to a tolerance of $\pm 3,3\text{ rev/sec}$ (200 r/min) at $66,7\text{ rev/sec}$ (4000 r/min) speed and $\pm 8,3\text{ rev/sec}$ (500 r/min) at 200 rev/sec ($12\ 000\text{ r/min}$).

If larger slurry volumes are needed, an alternate method for slurry preparation is found in Annex A of ISO 10426-2:—. As required, the density of the cement slurry can be determined by methods found in clause 6 of ISO 10426-2:—.

5.3.2.2 Slurry conditioning

The cement slurry should be poured immediately into the slurry cup of an atmospheric consistometer for conditioning. The slurry cup should be initially at ambient temperature to avoid the possibility of thermally shocking temperature sensitive slurries. The slurry should be then heated to the desired test temperature up to 90°C (194°F). The thickening time schedule which most closely simulates actual field conditions should be followed.

5.3.3 Curing at atmospheric pressure

After conditioning the slurry is poured into the large hole at the outer portion of the tip of the ring mould, preheated at test temperature in oven. The small hole in the top of the mould is for venting air from the mould during filling. The mould is filled until the slurry exits the small hole. On some thicker slurry, the mould may need to be tapped or vibrated to ensure it is completely filled.

Once the slurry is poured, the mould is placed into a thermostated water bath at test temperature. Slurry is in contact with water during the entire test. After curing, the sample is cooled to below 77 °C (170 °F). Water entry will compensate for any inner shrinkage as long as the cement matrix is permeable. If the cement expands during the hydration period the outside diameter of the annulus will expand.

5.3.4 Curing at pressure above atmospheric

After conditioning the slurry is poured into the large hole at the outer portion of the tip of the ring mould, preheated at 80°C (176 °F) in oven. The small hole in the top of the mould is for venting air from the mould during filling. The mould is filled until the slurry exits the small hole. On some thicker slurry, the mould may need to be tapped or vibrated to ensure it is completely filled.

Place the mould in a preheated [80°C (176°F)] pressure vessel. Further heat the slurry to test temperature in the time required to take the slurry from a depth with 80°C (176°F) circulating temperature to test temperature (use appropriate schedules described in clause 7 of ISO 10426-2:— or a schedule designed to simulate a specific well's conditions). Some heating chambers may not be able to heat fast enough and in that case heat as fast as possible but minimize overshooting the test temperature. Maintain the slurry at test temperature until it is time to cool the chamber to 80°C (176°F). The time required to cool the equipment from elevated temperatures to 80°C (176°F) will vary. The pressure applied can simulate bottom hole conditions, if desired.

5.3.5 Test Period

The test period is the elapsed time from subjecting the sample to temperature in the atmospheric consistometer to the time of final measurement to the end of curing time.

5.4 Measurement and Calculations

Before curing, in atmospheric bath or in pressure vessel, an initial measure is taken with a micrometer for calibration of the annular ring. The micrometer is opened and placed on the smooth, flat surface beside the mould to measure the distance between the outside of the steel balls attached to each side of the split on the expandable ring, with the spacer block in place if used.

After curing measurement is taken in the same manner as the initial measurement. If spacer block is used, carefully remove it. (important, do not expand the outer expandable ring while removing the spacer block). The

distance between the two steel balls is to be measured with a micrometer with a precision of 0,01 mm (0,01 inch). This measurement must be performed very quickly (less than 15 minutes) to prevent the specimen from sufficient cooling and causing an erroneous measurement.

One can measure linear shrinkage or expansion with this test. This is noted as $\%_{le}$, a positive value indicates expansion, a negative value indicates shrinkage.

The percent linear shrinkage or expansion is calculated as follows:

$$\%_{le} = (d_f - d_i) \times 0,358$$

Where:

$\%_{le}$ = is the linear shrinkage or expansion of the cement sample, expressed in per cent (%)

d_f = is the final distance measurement after curing, expressed in millimetres (mm)

d_i = is the initial distance measurement, expressed in millimetres (mm)

Alternatively, the percent linear shrinkage or expansion can be calculated as follows:

$$\%_{le} = (d_f - d_i) \times 9,095$$

Where:

$\%_{le}$ = is the linear shrinkage or expansion of the cement sample, expressed in per cent (%)

d_f = is the final distance measurement after curing, expressed in inches (in)

d_i = is the initial distance measurement, expressed in inches (in)

The coefficient is calculated considering an I.D. of outer expansion ring of 88,9 mm (3,5 inches).

It is assumed the linear expansion is the change in circumference as measured by the change in space by the balls. The measure is chords and not arcs, but because the difference is small, it can be neglected because of the small distance between the measurement balls, no correction is needed for extrapolating from the initial measurement of the mould at room temperature to the measurement at the actual test temperature. The theoretical correction for the metal used in the moulds is only 0,005% for each 35°C (100 °F) above the initial room temperature measurement. The result shall be reported with corresponding test period.

6 Determination of bulk shrinkage or expansion under impermeable condition: membrane test

6.1 General information

The purpose of this test method is to measure the bulk expansion or shrinkage when the cement is unable to imbibe water or gas. This is commonly the case when the cement is placed within impermeable boundaries in a well.

6.2 Apparatus

6.2.1 Membrane

Impermeable flexible membrane which is sealed by tying a knot at the top (Figure 5). The material of membrane must not react in high pH environment and should be capable to contain at least 150 ± 30 mL of slurry volume.

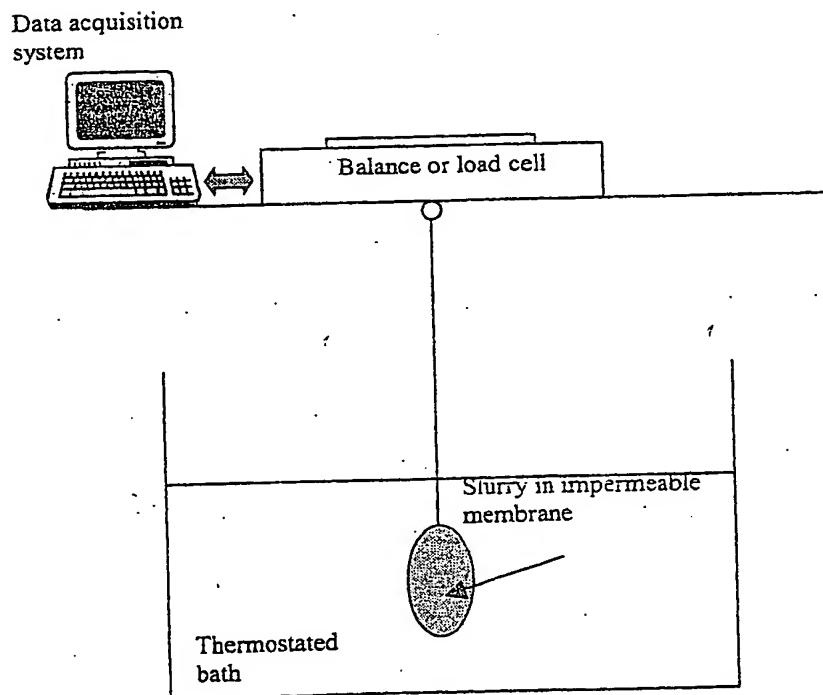


Figure 5

6.2.2 Water curing bath

A curing bath or tank having dimensions suitable for the complete immersion of the filled membrane in water and maintainable within $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$) of the prescribed test temperatures shall be employed. The water shall be distilled or deionized and degassed.

6.2.3 Temperature measuring system

The temperature measuring system shall be calibrated to an accuracy of $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$). Calibration shall be no less frequent than monthly. The procedure described in annex A of ISO 10426-2:— is commonly used. Two commonly used temperature measuring systems are:

6.2.3.1 Thermometer

A thermometer with a range including $21\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$ ($70\text{ }^{\circ}\text{F}$ to $212\text{ }^{\circ}\text{F}$) with minimum scale divisions not exceeding $1\text{ }^{\circ}\text{C}$ ($2\text{ }^{\circ}\text{F}$) may be used.

6.2.3.2 Thermocouple

A thermocouple system with the appropriate range may be used.

6.2.4 Electronic scales

Electronic scales with precision of $0,01\text{g}$ shall be used (preferred with data acquisition interfaced with computer)

6.3 Procedure

6.3.1 Preparation and placement of slurry

6.3.1.1 Slurry

Test samples should be prepared according to clause 5 of ISO 10426-2:—. The mixing device should be calibrated annually to a tolerance of $\pm 3,3\text{ rev/sec}$ (200 r/min) at $66,7\text{ rev/sec}$ (4000 r/min) speed and $\pm 8,3\text{ rev/sec}$ (500 r/min) at 200 rev/sec ($12\text{ }000\text{ r/min}$).

If larger slurry volumes are needed, an alternate method for slurry preparation is found in Annex A of ISO 10426-2:—. As required, the density of the cement slurry can be determined by methods found in clause 6 of ISO 10426-2:—.

6.3.1.2 Placing slurry in membrane

Condition the slurry in the atmospheric consistometer (see 5.2.5) for a period of 20 minutes ± 30 seconds. The bath temperature shall be maintained at $27 \pm 1,7^\circ\text{C}$ ($80 \pm 3^\circ\text{F}$) throughout the stirring period.

The slurry is poured into an impermeable flexible membrane, which is sealed by tying a knot at the top. Make sure the knot is absolutely tight, so no water from the bath can access the slurry during the test.

The volume of slurry shall be 150 ± 30 mL.

Settling of slurry must be avoided with properly optimized slurry composition.

Extreme care is taken so that no air is entrapped in the slurry or at the top of the membrane.

In particular, when one can see an expansion while cement has not yet reached the temperature of the bath (beginning of test), it may correspond to thermal expansion of the air, which has been trapped. In this case the test is invalid and must be repeated.

6.3.2 Curing

Once membrane has been prepared it is placed in a fine net and suspended to the balance hook and immersed in thermostated water bath at test temperature.

Attention shall be paid to avoid that air is attached to the external surfaces of the membrane or net.

The water evaporation in the bath is not critical at 35°C (95°F). It becomes significant at higher temperatures.

A reference level is chosen for the water bath and water regularly added at the same temperature $\pm 2^\circ\text{C}$ ($\pm 3^\circ\text{F}$).

6.4 Measurement and Calculations

The pseudo weight, as measured by the balance, is the weight of the membrane containing cement minus the buoyancy force acting on it ($W - B$). B is equal to the volume of the membrane containing cement multiplied by the specific gravity of water ($1,00 \text{ kg/L}$).

The evolutions of the sample volume and temperature are recorded during the test. Data are recorded every 10 minutes. An increase of the pseudo weight corresponds to a decrease of the volume (shrinkage).

After a certain period of time, the hardened cement is removed from the membrane and weighed. This set up is used to measure bulk shrinkage or bulk expansion.

Because the membrane test can result in either a decrease or an increase of the external volume, the volumetric change measured with the membrane is called percent bulk change and noted as $\%_{le}$.

To calculate the initial and final volumes, and then the shrinkage or expansion, one assumes that the weight and volume of the membrane and the net are very small.

Therefore a positive value corresponds to a bulk expansion, while a negative value corresponds to a bulk shrinkage.

The percent shrinkage or expansion is calculated as follows:

$$\%_{le} = 100 \times (V_f - V_i) / V_i \quad (3)$$

Where:

$$V_i = W - (W - B_i) \quad (4)$$

$$V_f = W - (W - B_f) \quad (5)$$

Where:

V_i = initial volume of the cement slurry, expressed in millilitres (mL)
 V_f = final volume of the cement slurry, expressed in millilitres (mL)
 W = weight of cement slurry, expressed in gram (g)
 B_i = initial volume of the membrane containing cement slurry, expressed in millilitres (mL)
 B_f = final volume of the membrane containing cement slurry, expressed in millilitres (mL)

The test period is the elapsed time from subjecting the sample to curing in the atmospheric consistometer to the time of final measurement to the end of curing time.



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SPE/IADC 79911

Real-Time Cement Expansion/Shrinkage Testing Under Downhole Conditions For Enhanced Annular Isolation

Virgilio C. Goboncan, SPE; Robert L. Dillenbeck, SPE; BJ Services Company

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This paper was prepared for presentation at the SPE/IADC Drilling Conference held in Amsterdam, The Netherlands, 19-21 February 2003.

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Abstract

Ongoing problems with sustained annular gas pressure on producing wells worldwide is a prime indicator that more work is needed in understanding how cement system design can impact long-term annular isolation. One important area is the relative expansion or shrinkage of cement systems as they hydrate in the annulus of a well.

Most Portland cement systems undergo chemical and bulk shrinkage as they set. A number of chemical materials have been developed that can be added to cementing systems to overcome this shrinkage, and in some cases, even impart a degree of expansion. However, the process whereby the degree of shrinkage or expansion is measured has almost universally been some type of a single point test, performed at ambient conditions. This is typically the case even if the cement sample was first hydrated under downhole conditions.

The authors detail new methodology and test equipment that allows for the tracking of cement shrinkage and/or expansion real-time, under downhole conditions of pressure and temperature. The system allows for the application of curing pressures in either an aqueous or non-aqueous environment. Data acquisition is totally computerized and can be maintained for whatever time interval is required, without exposing the hydrating cement to surface conditions. With this new technology, the authors will detail how cement expansion and shrinkage can be significantly different under downhole conditions of pressure and temperature than when measured at ambient conditions. It has also been observed by the authors that cement expansion or shrinkage is a dynamic process that may take many hours or even days before any significant degree of volumetric stability is achieved.

Finally, a successful field application of the new testing methodology will be presented as validation for the transfer of this new technology from the laboratory to the real world of oil and gas well cementing.

Introduction

Service companies and operators alike have become more aware of the many factors that can influence long-term zonal isolation in a cemented annulus. It is now apparent that a significant change in the physical volume of the set cement in an annulus, be it an excessive reduction (shrinkage), or even excessive increase (expansion), can play a major role in the determination of success or failure for long-term annular isolation. In order to better quantify these cement hydration volume changes, the authors have seen the industry progress from days when positive expansion was tested simply by observing if the setting cement would expand enough to break a glass bottle, to their newest micro-processor controlled expansion testor.

Over the years, many skilled in the art of oil and gas well cementation have extensively researched the shrinkage that typically occurs when a Portland cement-based annular sealant hydrates, and undergoes a chemical transition from a liquid slurry state to the final set state, which will hopefully effect a long-term seal of the well's annulus. The fact that most typical Portland cement systems will exhibit some degree of shrinkage during this "setting", or hydration process, has been well documented in the literature, and for the most part is now accepted within the industry. The deleterious effects of cement shrinkage on annular isolation have also been well documented. This acceptance is in spite of the fact that there have been a wide variety of tests and devices developed in an effort to quantify both the type, and amount of shrinkage that occurs, without the oil and gas industry ever standardizing testing for cement volumetric changes during hydration.

It is this same generally accepted "fact" about cement shrinkage that has resulted in the development of a number of "solutions" to the problem. These solutions generally fall into one of two broad categories. The first category would be various chemical materials that interact with the Portland cement blend as it hydrates to produce expansion sufficient to counteract the natural shrinkage that still typically occurs. The second category would be either gaseous or mechanical energizing materials that can be added to the cement blend, and that will expand during cement hydration to counteract volume losses. The authors noted that typically, the same types of testing devices used to previously document cement shrinkage were often used in attempts to quantify cement expansion. Even today, these same devices are generally used whenever a need exists to quantify both the presence and amount of expansion present in a given application for a

specific cement blend. The authors themselves are quite familiar with most of the more widely used expansion test devices, and have had occasion to use a number of them over the years. However, they felt that all the common testing methods and/or devices that they were familiar with suffered from one or two common deficiencies.

The first deficiency is that most of the expansion test devices are a single point test. In other words, the tests yielded a single value for expansion or contraction at a given point in time. It has been the experience of the authors that cement volumetric change during hydration is a very dynamic event that can continue over a long period of time. Because of this, there is concern about how well such a "single point" type of a test could capture the entire expansion event.

The second deficiency that the authors identified was that almost all of the tests currently in use lacked the ability to actually physically quantify cement linear expansion in an uninterrupted downhole environment. What this meant was that with many current testing methods, the samples are simply cured under atmospheric, or near atmospheric pressure, and at temperatures limited to the boiling point of water under ambient conditions. Further, even if the cement sample is cured under conditions of downhole temperature and pressure, when it is time to take a "single point" measurement, the sample has to be removed from the temperature and pressure controlled autoclave, and brought to ambient conditions before any physical measurements of expansion can be performed.

The authors note that such drastic changes in temperature and pressure will most certainly have at least some negative impact on the data from such tests. If the samples are not cured under downhole conditions, the reaction of the cement and expansion additive may not be the same as occurs under surface conditions. On the other hand, even if the samples are cured under downhole conditions, one still has to be concerned about the effect of cooling and depressurizing the sample to ambient conditions before any measurements can be made.

Further, the authors note that with current tests possessing these inherent deficiencies, the data gathered on the performance of various expansive additives might be flawed when compared to what happens downhole. Based on the authors' research, it would seem that others like Baumgart¹ have likewise concluded that expansive materials may likely respond differently, depending upon, among other things, curing temperature and pressure. As early as 1984, when studying the effect of cement shrinkage or expansion on cement bonding, Parcevax² wrote of a need for new equipment that "must be designed so that both curing and testing of the cement samples are performed under the same conditions without having to manipulate the samples". Finally, some 18 years later, the authors believe that they have developed a testing device, which for real-time direct determination of cement volumetric change during hydration, will do just what Parcevax² was proposing.

Current Testing Methods

At this point, the authors feel that a short review of some of the commonly used expansion testing devices currently in use is in order. This review should allow the reader to be able to better understand and then differentiate between the current

expansion testing devices and the expansion-testing device developed and presented as the subject of this paper.

One of the oldest, but still commonly used test method is the ASTM expansion test, C-151-00. In this test, a 1 inch x 1 inch x 1 1/4 inch mold is used to cast a cement sample, typically under ambient laboratory conditions. Upon initial set, the sample is removed from the mold, and then the length of the sample is precisely measured (usually to +/- 0.001 inch). This value is typically used as the zero or starting point. After the recording of the starting point length, the sample can then be subjected to various conditions of heat, humidity and sometimes pressure (though typically, the pressure is still maintained at ambient levels) for a desired period of time, before the length measurement is repeated to record any net change. A positive net change would indicate linear expansion, while a negative net change would indicate cement shrinkage. By using both data points, a percentage linear expansion or shrinkage is typically then calculated. Obviously, as previously noted, this type of a test does not typically perform the initial curing of the sample at bottom hole conditions of temperature and pressure. Further, even if the sample is later exposed to temperature (after initial set), it must then be re-stabilized at ambient temperature before a meaningful data point can be recorded. Any attempt to record a data point at a temperature significantly different from that at which the original zero, or starting point was recorded, would introduce significant error due to thermal expansion or (with lowered temperature) contraction. Finally, as discussed previously, this test yields only single point in time type data. Any dynamic expansion or shrinkage that occurs between the times measurements are taken, goes undetected. Figure 1 is an example of the ASTM expansion testing device.

Another widely used quantitative test of cement expansion is often called the "split-ring" test. In this test, a split, expandable ring is typically placed on top of a flat metal plate, with another "top" plate held in place with a screw. The split in the ring is usually held lightly with light spring pressure. Once the mold is assembled and liquid cement slurry is poured into the mold through a small hole in the top plate, air is removed by gently tapping the mold. The mold containing the cement is then placed into an autoclave to allow for initial cement set.

After the cement has achieved an initial set, a measurement across the two measurement points that span the "split" in the ring is made. The whole assembly is then placed back in the autoclave for curing of the cement for the desired time under correct temperature and pressure conditions. Typically, the hydraulic medium used to apply this pressure is fresh water, and this provides an additional source of water to the cement during hydration. Once the desired test time period passes, the assembly is cooled and de-pressurized back to ambient conditions and then is once again removed from the autoclave and a second measurement made. Any net positive expansion that has occurred over the time of the test can then be quantified by a second measurement, which shows any increased distance across the "split" in the ring. This increase can then be used to calculate a percent total linear expansion. A split-ring testing device is shown in Figure 2.

A similar test to the "split-ring" procedure utilizes a "split-cylinder" which is taller than the "split-ring" and, therefore,

typically allows for three points of expansion measurement along the length of the cylinder, instead of just the one used on the "split-ring". These three measurements are normally then averaged to determine the amount of expansion occurring over the course of the test. In all other ways, the two tests are very similar. In both cases, the single point-in-time test result only captures net expansion present at the time the measurements are taken, as also occurs with the ASTM test.

Also, as in the case of the ASTM expansion test, the cured cement samples from the "split-ring" and "split-cylinder" must undergo the stresses and potential dimension altering effects imposed by a return from autoclave conditions to ambient conditions, before any measurements can be made, and net expansion calculated. Also, in either the ASTM, or "split-ring, split-cylinder" type expansion tests, before calculations of total net linear expansion can be completed, manual human measurements must be taken, and thus, the possibility of human error is also introduced into the process. These errors can take the form of either the mathematical calculations phase of the process, or even if that part of the process is computerized, human error can occur in the physical act of taking the measurements with very sensitive manual measuring devices necessitated by the process. Human error can also come into play if the measurements are made before the samples have returned to the same temperature as when the preliminary measurements were made.

Finally, there is one type of test that does attempt to quantify the entire historical expansion/shrinkage process for given cement systems through a continuous process. The dilatometric method places a cement slurry sample in a container such that the permeable container holding the cement slurry sample is itself installed in a larger pressure vessel. This larger pressure vessel typically uses water as the hydraulic medium, and the cement slurry sample contained within the pressure vessel is exposed to this water in the vessel. Once the cement slurry sample is placed inside the pressure vessel, the pressure and temperature inside the pressure vessel are stabilized at those conditions desired for the test. Once these conditions have been reached at the start of the test, the temperature and pressure are very carefully maintained at the same level. By the use of very precise, automatic volume measuring devices, these very small pressure maintaining volume adjustments could be captured real-time with computerized data acquisition equipment to give a continuous plot of the pressure vessel volume changes. The volume of water that must be added or removed from the pressure vessel for pressure maintenance after the initial stabilization of temperature and pressure is typically thought by some to directly reflect corresponding expansion or shrinkage of the cement system as it hydrates.

Parcevaux² utilized this type of test in an attempt to quantify real-time cement expansion or shrinkage under undisturbed downhole conditions. Unfortunately, upon further analysis, it is the opinion of the authors of this paper, as well as others³ that though this type of test does succeed in generating data without the need to manipulate the sample at ambient conditions, it fails in one critical area. Since the external dimensions of the cement sample are never directly measured, the actual dimensions of the cement sample can actually grow, shrink, or remain the same without being detected. In

fact, as long as the cement is still hydrating and some internal permeability remains, the recording of water volume changes within the pressurized vessel may only be indicative of water being consumed or expelled during the cement chemical hydration process, (often referred to as chemical shrinkage by many) and not in any actual change in the physical dimensions of the cement sample. Though the consumption of water within a hydrating cement sample most certainly impacts important issues such as pore pressure reduction, and possible gas migration, the authors of this work are primarily interested in the actual changes in the physical dimensions (expansion/shrinkage) of cement samples under downhole conditions. For this reason, another testing device was developed that they believe allows for better direct determination of the actual real-time cement bulk volume change during hydration under downhole conditions of temperature and pressure, without the need for sample manipulation.

New Testing Device

The device presented in this work was developed in an effort to allow for a cement system to undergo initial curing and subsequent hydration under uninterrupted downhole conditions of temperature and pressure, while at the same time, continuously and directly monitoring dimensional changes resulting in linear expansion or shrinkage. Further, once the cement test samples are brought to downhole conditions of temperature and pressure, these conditions are maintained via microprocessor controls for the duration of the test. The samples are never returned to ambient conditions of temperature and pressure until such time as the test is terminated. This prevents errors from occurring that might otherwise be caused by changes in temperature and pressure. All real-time data acquisition is via microprocessors and can be stored in electronic data files for subsequent retrieval and analysis. Given the high degree of automated control, data acquisition, and expansion calculation used in the device, the possibility of human error in the testing process is for the most part removed. Further, the design of the device leads for a high degree of repeatability in tests.

From a functionality point of view, the device consists of a special microprocessor controlled high-pressure, high-temperature (HPHT) autoclave, which is used to maintain downhole conditions of temperature and pressure on the cement sample during the test. A specially designed expansion/contraction cement mold has been developed to allow direct measurement of the cement sample under downhole conditions. The mold is designed in such a way that after it is filled with slurry and fitted with a metal top plate, it allows for cement expansion or contraction (shrinkage) during hydration in three distinct axis, x, y, and z (vertical). This special mold utilizes spring-loaded resilient side seals that allow for either expansion or contraction of the mold in either the x, or y-axis. Any expansion of the cement in the x, or y-axis will result in an increase in the external dimension of the mold, and also result in the compression of the springs that hold the mold together. Depending upon the exact placement of the mold in the test vessel, the total expansion available in the mold (before bottoming out the springs) will generally equate to between 9% to 14% total linear expansion. Cement

expansion along the z (vertical) axis is basically unrestricted, since the metal top plate that is placed over the cement slurry in the mold is not restrained, and is free to be lifted by expanding cement.

In the event of cement shrinkage, two sacrificial studs are placed on the interior of the mold before adding the cement slurry such that the curing cement will "lock" onto the heads of the studs and via the stud body, pull on the mold exterior and compress the resilient seals, resulting in a reduction in the molds external dimensions.

The special HPHT autoclave is fitted with one or more highly sensitive, HPHT, electromagnetically loaded, linear displacement transducers (LDT). Once the special mold is assembled and filled with cement slurry, it is located into the special autoclave vessel such that the spring-loaded LDT rests against the side of the cement mold. The autoclave is then closed and filled with fluid to transmit pressure and temperature to the cement sample for the duration of the test. Though typically fresh water is used for this hydraulic medium, the unit is capable of being switched over to use a high-grade mineral oil for this purpose, which will be discussed further at a later point in this paper. Figure 3 illustrates the design of the special cement mold used in this device, and how that mold is located inside the HTHP autoclave in relation to the HTHP LDT.

After sealing the pressure vessel, the temperature and pressure within the vessel are typically ramped up to bottom hole conditions over a time consistent with an appropriate API schedule that would be used for the determination of cement compressive strengths for the depth and temperatures of a well that the cement system would be applied in. Once the final bottomhole pressure and temperature have been achieved, and the test device has stabilized, the LDT reading is set as the zero point for the duration of the test. Any subsequent movement of the LDT above or below this point is interpreted as expansion or shrinkage of the cement sample. All pressure, temperature, and LDT data is automatically and continuously fed into a computerized data acquisition unit (DAU). With the temperature and pressure of the device held constant, any expansion or shrinkage of the actual cement sample is detected by the LDT and fed real-time into the DAU, where it is automatically converted to a net percent linear expansion. At any point during the testing of a cement system, the DAU can be queried and results up to that time displayed in a graphical format showing percent linear expansion or shrinkage vs. time, along with the pressure and temperatures maintained during the test. Upon completion of a test, the entire history of the test can be recalled and displayed on a computer or printed graphically.

Since all data generated by this device is produced at steady bottom hole conditions, the authors are confident that possible errors introduced with other test methods that require transitioning the cement samples to ambient conditions before being measured are avoided. Further, unlike the dilatometric method, the changes in actual dimensions are measured, instead of just the volume of fluid either consumed or expelled by the hydrating cement.

Example Test Data

With the capabilities afforded by the new expansion test device, the authors ran several different series of tests with known cement expansion additives, over different time periods. One of the first things noted was confirmation of the theory expressed previously by others^{1,2,4,5} and long suspected by the authors, that cement expansion is a dynamic process that is not always linear and proceeds over an extended period of time. In a test shown in Figure 4, a slurry containing no expansion additive actually exhibited volume changes which at one point, had the test been stopped at that time, would have indicated cement expansion, and yet at another point in time would have shown net cement shrinkage. In other tests, such as the one illustrated in Figure 5, it was noted that with a different expansion material, maximum expansion did not occur until about four days into the test. Obviously, if a single data point was recorded (from a conventional expansion test) on the same slurry, and for a time period shorter than four days, the full benefit of the particular expansion additive might have been missed.

Once the authors were familiar with the type of data they could obtain with the new expansion test, the next logical step was to compare test results from the new device with results previously obtained with the conventional expansion tests. In the middle of just such a test sequence, a unique field challenge was presented, which was to ultimately allow the authors to solve a challenging field cementing situation, and at the same time, validate the usefulness of the new expansion test device.

Field Application

A unique cementing challenge was presented to the authors after the new expansion test device had been in use in the laboratory for a short time. In a very prolific HTHP gas field, an operator experienced sustained gas pressure build up in the annulus of many of the wells at surface. In many instances, the gas appeared at surface in the annulus of the wells very quickly after the completion of primary cementing operations, and at times, even before the wells could be completed and put on production. Among various steps being employed to attempt to stop the gas migration up the annulus, the operator had been using 5% by weight of cement (BWOC) of a chemical expansion additive, in almost all of its primary cement jobs. Unfortunately, gas was still present on the backside of many of the wells this operator was completing, and so further examination of the problem was obviously necessary. During various cement tests conducted on the slurries in use in this particular field, the authors ran a conventional "split-ring" type expansion test to quantify the amount of linear expansion produced by the 5% BWOC expansion additive. Results of this test showed that under the single data point "split-ring" test, the system exhibited 5.72% net linear expansion after 14 days. Though an impressive figure, the authors wondered, given other testing they were doing with their new expansion test device, if the values achieved with "split-ring" test were truly indicative of the results that were actually taking place in the operators' wells. To find out, they ran another test with 5% BWOC expansion additive in their new downhole cement expansion test device.

Though the authors anticipated some amount of difference in the data produced by the downhole, real-time tests, they were still not prepared for the magnitude of the difference that they recorded. Instead of the 5.72% linear expansion produced in 14 days by the "split-ring" expansion test, the new expansion test device showed that when undisturbed at downhole conditions of temperature and expansion, the subject cement system expanded to almost 1.1% linear expansion, in 47 hours. In fact, the final linear expansion produced under downhole conditions was not quantifiable, due to the fact that the cement expansion had bottomed out the mold retaining screws, effectively reaching the limits of the device to record further expansion after about 28 hours. In order to check the accuracy of their test data, a new sample of the cement system was prepared again, and the test repeated, with the same results. Figure 6 shows the resulting linear expansion vs. time plot for these tests. Another troubling aspect of these tests surfaced when the cement samples were removed from the autoclave after the end of testing. Expansion had actually been so great that visual inspection of the set cement showed the samples had literally "grown" out of the mold. The entire cement sample was also covered with a series of what appeared to be expansion induced fractures.

After reproducing the large linear expansion of the 5% system under downhole conditions, the authors discussed the results with others familiar with the chemistry involved in the expansion additive being used. A question that developed from these conversations regarded the availability of additional fresh water downhole in the annulus of the subject gas wells. The concern was that even though the new expansion device used fresh water as the hydraulic medium, and hence gave hydrating cement samples an ample supply of additional water, in the "real world" of dry gas wells, the same cement system might not have such "extra" water available for use in the expansion process. To test if the additional water was indeed necessary in order for the system to achieve such high levels of expansion under downhole conditions, a modified test was performed. In this test, special care was taken to use extra grease in order to seal as completely as possible the upper surface of the mold where top plate rests on it. Unfortunately, this temporary seal most likely did not survive the HTHP environment of the autoclave, and once significant expansion occurred in the vertical axis, the plate was lifted high enough to break any remaining seal and allow direct cement-to-water contact.

At this point, the decision was made to retrofit the HTHP autoclave to be able to use a high-grade mineral oil instead of water as the hydraulic medium in the expansion tests. By using the mineral oil instead of water as the hydraulic medium inside the autoclave, no water other than that initially used to prepare the cement slurry would be available to the cement system for the duration of the test. When the expansion test with 5% BWOC expansion additive was repeated with the mineral oil instead of water in the HTHP autoclave, the rate of expansion was somewhat slower and the total somewhat lower than when water had been used, but total linear expansion still exceeded 7%. Figure 7 illustrates the results of this test done with mineral oil as the hydraulic medium.

After seeing such a large amount of linear expansion with either water or oil in the HTHP autoclave, the authors were

faced with a true paradox: while most in the industry have been concerned about excessive cement shrinkage causing a loss of annular isolation, the authors became concerned about the possibility that excessive expansion might possibly be causing at least some of the problems with annular isolation in the subject wells. The authors understood quite well that in their expansion test, no confinement approaching the amount present in a typical well is placed on the curing cement. However, they also noted others who had at least expressed concern about excessive cement expansion should it occur downhole, and the negative effect it might have on annular isolation. Moran³ discusses at least the possibility of excess cement expansion causing annular isolation problems, while Baumgarte¹ points out that excessive downhole cement expansion might lead to the creation of micro annulus between the casing and the cement sheath. Ghofrani⁶ noted that in order to obtain enhanced bonding, cement expansion needed to occur during the "nearly hardened phase" of cement hydration. Because of the hazards associated with expansion in this phase of the cement hydration, he recommended using "small quantities" of additives that promote cement expansion.

Based upon the large expansion the authors recorded being generated under downhole conditions with 5% BWOC of the expansion additive, and coupled with the indications in the published literature that indicated possible deleterious effects from excessive downhole cement expansions, additional expansion testing was carried out with reduced loadings of the expansion additive. Ultimately, a loading of from 0.75% to 1.0% BWOC was selected, based on test results. This loading range gave repeatable results where the cement was either volumetrically stable, or expanded slightly (less than about 3%) before the cement volume stabilized, as can be seen in Figure 8.

This new loading for the cement expansion additive was recommended to the field, and the first of several wells were cemented with the reduced expansion additive loading. It should also be noted that other than the reduced expansion additive loadings, the rest of the slurry composition remained fundamentally the same as the system used in previous wells that had exhibited gas at surface in the annulus. Reports back from the field indicated that the first three wells cemented with the reduced expansion additive loading were gas free in the annulus at surface. Even after the first of the three wells had been completed and hydraulically stimulated, annular isolation apparently remained intact, and no annular gas was detected at the surface. Given the initial success with the controlled expansion cement system, all subsequent wells have continued to be cemented with the same type systems and results continue to be favorable.

Though insufficient data exists to pinpoint an exact cause for the initial loss of annular isolation in these wells, the authors feel that the success achieved by reducing downhole cement expansion may yield some strong clues. They believe that there exists a very high likelihood that either the cement matrix permeability might have become excessively high due to expansion induced micro fractures, or perhaps preferential cement expansion towards the borehole walls created radial micro annuli sufficient for a loss of annular isolation.

Conclusions

From the investigations and results discussed in the paper, the authors made the following conclusions:

1. Cement volumetric change during hydration is dynamic and may not stabilize for many hours or even days after initial set has occurred.
2. Common expansion or shrinkage tests that only produce a single data point, for a single point in time, may not capture the final stabilized volume for a given cement system. This can lead to the application of non-optimized expanding cement systems.
3. Testing of actual cement bulk volume changes under downhole conditions of temperature and pressure may yield very different results than those obtained under ambient laboratory conditions.
4. Under certain downhole conditions, excessive cement volume expansion can contribute to a loss of annular isolation.
5. By testing cement systems under downhole conditions of pressure and temperature, cement expansion additives can be optimized to yield a shrinkage compensated system with only enough controlled expansion to enable long-term annular isolation.

Nomenclature

- ASTM = American Society for Testing and Materials
 HTHP = high temperature high pressure
 LDT = linear displacement transducer
 DAU = data acquisition unit
 API = American Petroleum Institute
 BWOC = by weight of cement

Acknowledgements

The authors would like to acknowledge the assistance of Mr. Murray Rogers, Mr. Scott Bray, Mr. Thomas Heinold, and Dr. Gerald Braun, all with BJ Services. The authors would also like to thank Mrs. Doris Porter, and Ms. Issa Mendez of BJ Services Company for their assistance in reviewing this paper, and the management of BJ Services Company, for their permission to prepare and present this paper.

References

1. Baumgarte, C. et al: "Case Studies of Expanding Cement To Prevent Microannular Formation" paper SPE 56535 presented at the 1999 SPE Annual Technical Conference and Exhibition, Houston, Texas, October 3-6.
2. Parcevaux, P.A. and Sault, P.H.: "Cement Shrinkage and Elasticity: A New Approach for a Good Zonal Isolation" paper SPE 13176 presented at the 1984 SPE Annual Technical Conference and Exhibition, Houston, Texas, September 16-19.
3. Moran, L.K. et al: "Cement Expansion: A Laboratory Investigation" paper SPE 21685 presented at the 1991 SPE Production Operations Symposium, Oklahoma City, Oklahoma, April 7-9.

4. Root, R.L. and Calvert, D.G.: "The Real Story of Cement Expansion" paper SPE 3346 presented at the 1971 SPE Rocky Mountain Regional Meeting, Billings, Montana, June 2-4.
5. Sabins, F. L. and Sutton, D.L.: "Interrelationship Between Critical Cement Properties and Volume Changes During Cement Setting" paper SPE 20451 presented at the 1990 SPE Annual Technology Conference, New Orleans, Louisiana, September 23-26.
6. Ghofrani, R. and Plack, H.: "CaO and/or MgO-Swelling Cements: A Key for Providing Better Annular Sealing?" paper SPE/IADC 25697 presented at the 1993 SPE/IADC Drilling Conference, Amsterdam, The Netherlands, February 23-25.

Figure #1

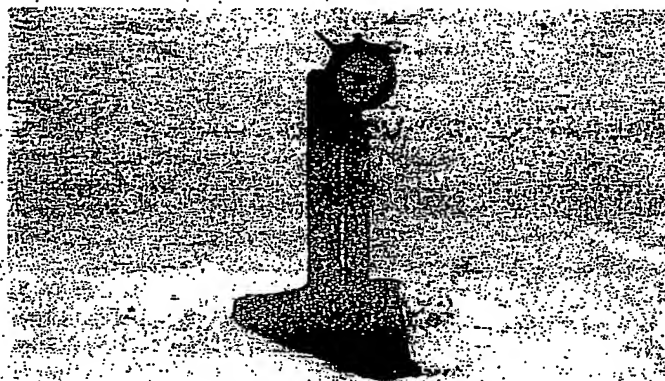


Figure #2



Figure # 3

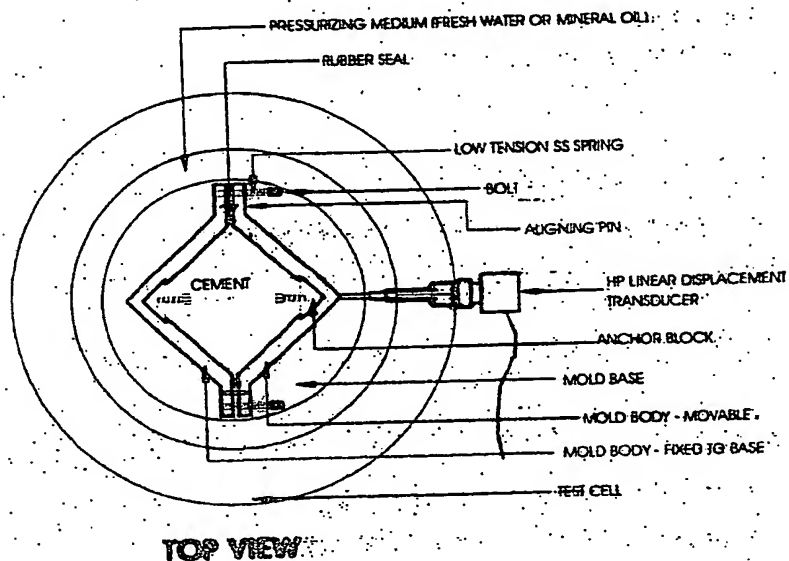


Figure # 4

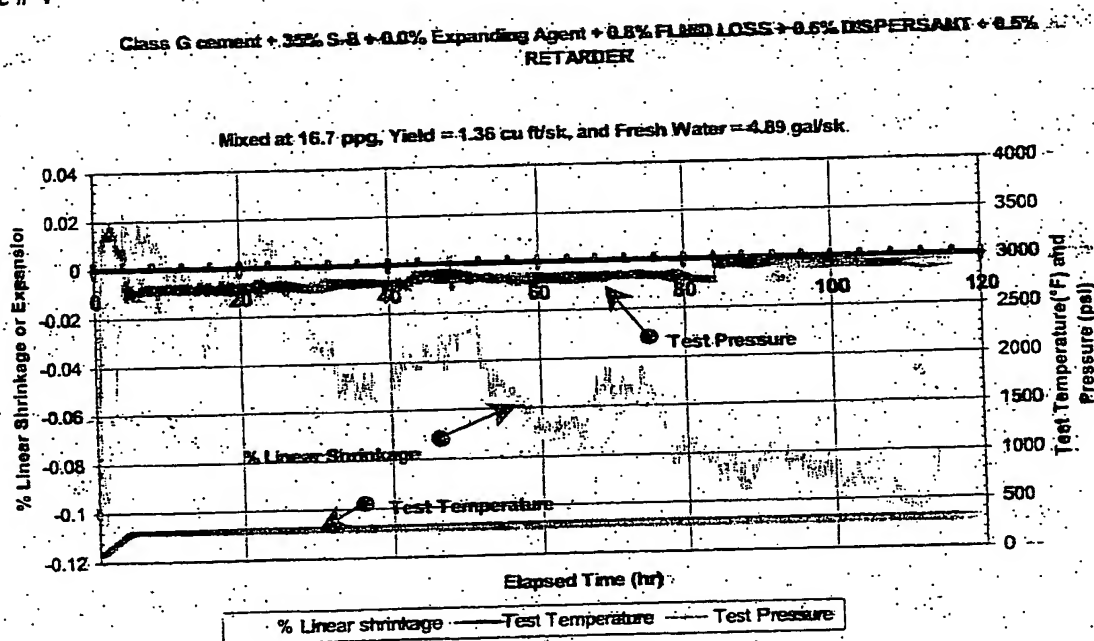


Figure # 5

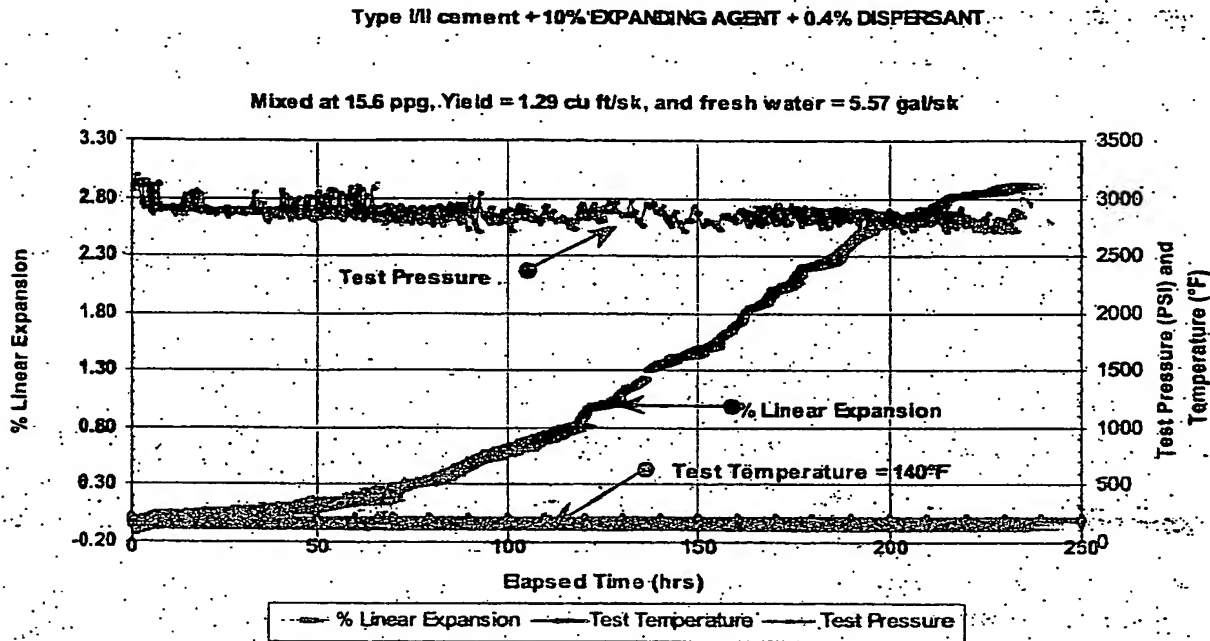


Figure # 6

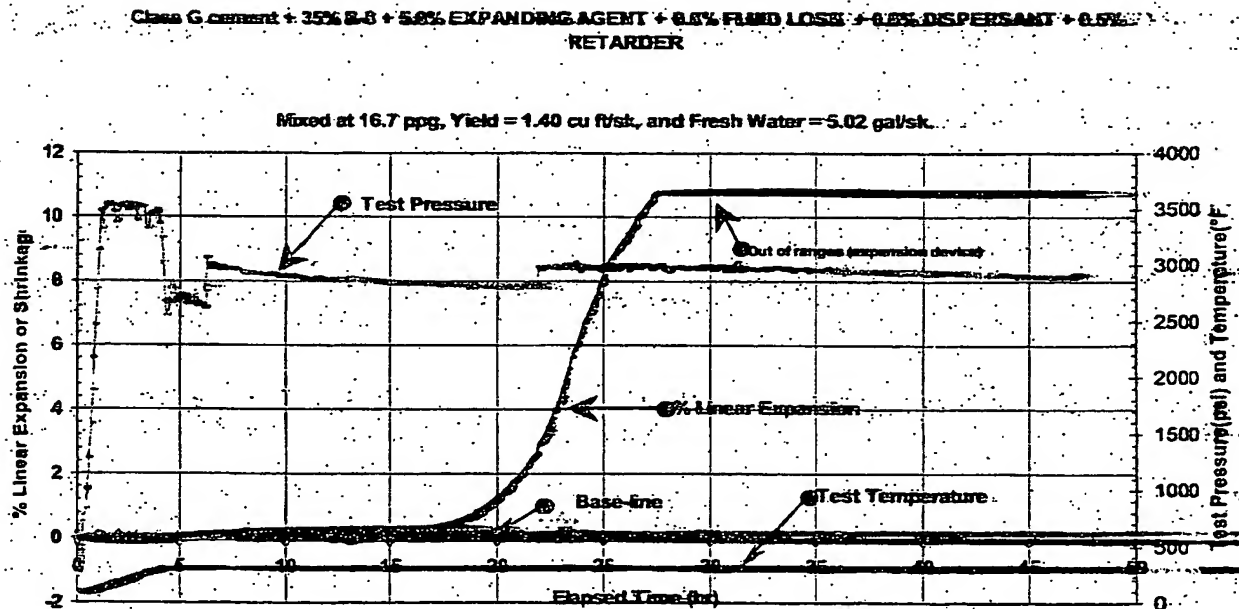


Figure # 7

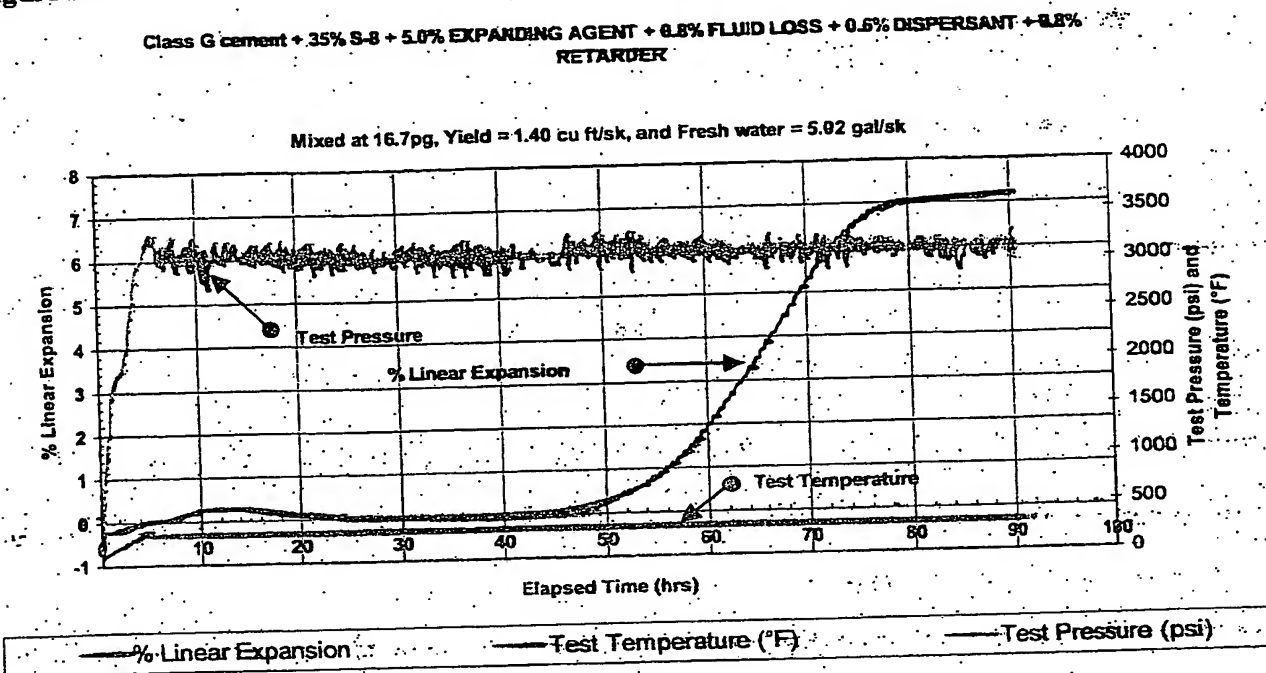
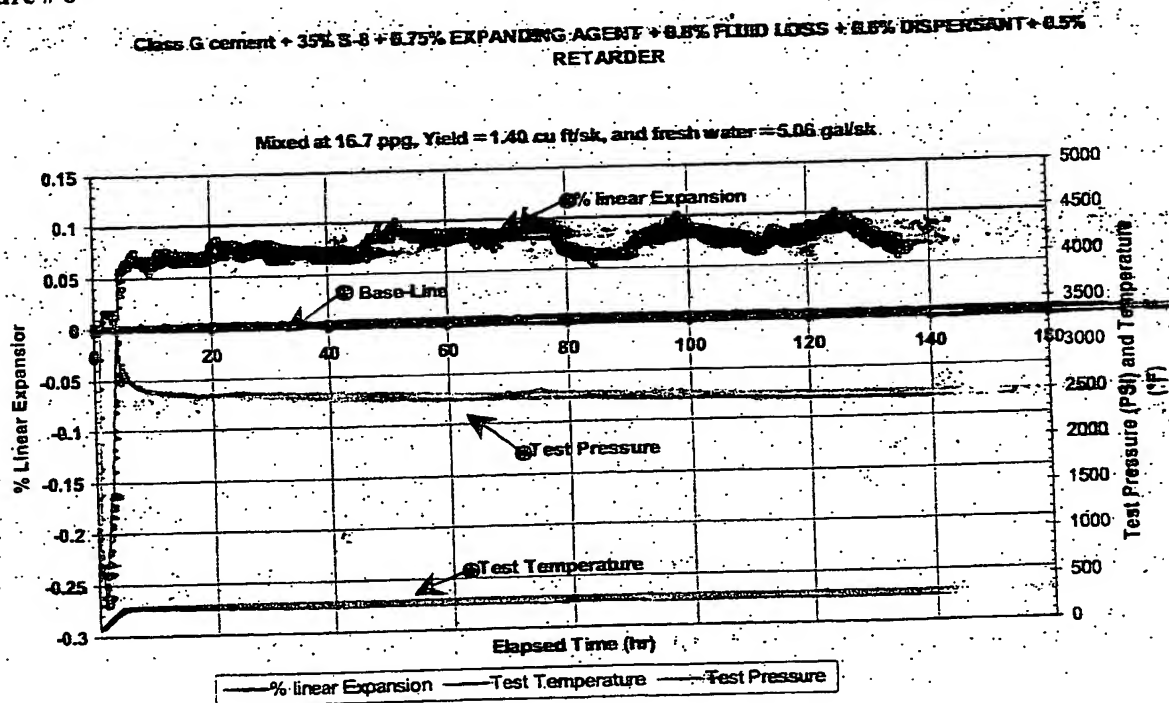
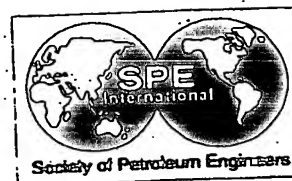


Figure # 8





SPE 64733

Why Oilwells Leak: Cement Behavior and Long-Term Consequences

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This paper was prepared for presentation at the SPE International Oil and Gas Conference and Exhibition in China held in Beijing, China, 7-10 November 2000.

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Abstract

Oil and gas wells can develop gas leaks along the casing years after production has ceased and the well has been plugged and abandoned (P&A). Explanatory mechanisms include channeling, poor cake removal, shrinkage, and high cement permeability. The reason is probably cement shrinkage that leads to circumferential fractures that are propagated upward by the slow accumulation of gas under pressure behind the casing. Assuming this hypothesis is robust, it must lead to better practice and better cement formulations.

Introduction, Environmental Issues

This discussion is necessarily superficial, given the complexity of the issue and attendant practical factors such as workability, density, set retardation, mud cake removal, entrapment of formation gas, shale sloughing, pumping rate, mix consistency, and so on. A conceptual model will be developed in this article to explain slow gas migration behind casing, but we deliberately leave aside for now the complex operational issues associated with cement placement and behavior.

In 1997, there were ~35,000 inactive wells in Alberta alone, tens of thousands of abandoned and orphan wells, plus tens of thousands of active wells. Wells are cased for environmental security and zonal isolation. In the Canadian heavy oil belt, it is common to use a single production casing string to surface (Figure 1); for deeper wells, additional casing strings may be necessary, and surface casing to isolate shallow unconsolidated sediments is required. As we will see, surface casings have little effect on gas migration, though they undoubtedly give more security against blowouts and protect shallow sediments from mud filtrate and pressurization.

To form hydraulic seals for conservation and to isolate deep strata from the surface to protect the atmosphere and shallow groundwater sources, casings are cemented using water-cement slurries. These are pumped down the casing, displacing drilling fluids from the casing-rock annulus, leaving a sheath of cement to set and harden (Figure 1). Casing and rock are prepared by careful conditioning using centralizers, mudcake scrapers, and so on. During placement, casing is rotated and moved to increase the sealing effectiveness of the cement grout. Recent techniques to enhance casing-rock-cement sealing may include vibrating the casing, partial cementation and annular filling using a small diameter tube.

Additives may be incorporated to alter properties, but Portland Class G (API rating) oil well cement forms the base of almost all oil well cements.² Generally, slurries are placed at densities about 2.0 Mg/m³, but at such low densities will shrink and will be influenced by the elevated pressures (10-70 MPa) and temperatures (35 to >140°C) encountered at depth.

The consequences of cement shrinkage are non-trivial: in North America, there are literally tens of thousands of abandoned, inactive, or active oil and gas wells, including gas storage wells, that currently leak gas to surface. Much of this enters the atmosphere directly, contributing slightly to greenhouse effects. Some of the gas enters shallow aquifers, where traces of sulfurous compounds can render the water non-potable, or where the methane itself can generate unpleasant effects such as gas locking of household wells, or gas entering household systems to come out when taps are turned on.

Methane from leaking wells is widely known in aquifers in Peace River and Lloydminster areas (Alberta), where there are anecdotes of the gas in kitchen tap water being ignited. Because of the nature of the mechanism, the problem is unlikely to attenuate, and the concentration of the gases in the shallow aquifers will increase with time.

This implies that current standards for oilwell cementing and P&A are either not well founded, or the criteria are based on a flawed view of the mechanism. This is not a condemnation of industry: all companies seek to comply with standards.³ Nevertheless, we believe that the AEUB Interim Directive 99-03⁴ is flawed with respect to gas leakage around casings. To rectify this, the mechanisms must be identified correctly. Practice can then be based on correct physical mechanisms, giving a better chance of success (though we do not believe

that the problem can be totally eliminated because of the vagaries of nature and human factors, despite our best efforts).

There is also need for better quality oil-well cement formulations that can resist thermal shocking. For example, leakage of fluids along thermal wells in cyclic steam operations in Alberta has proven a challenging problem for Imperial Oil.⁵ If poor quality or poorly constituted cement is used, high injection pressures, thermal shocking, plus non-condensable gas evolution lead to leakage behind the casing that could break to surface under exceptional conditions.

Finally, in production management for conservation purposes, zonal isolation is multiple-zone wells.⁶

There are initiatives to identify old leaking wells and undertake mitigating action in Alberta and Saskatchewan, the "orphan well" program of the AEUB, initiatives by the Petroleum Technology Alliance Centre in Calgary, and so on. This article is to try and clarify the mechanisms involved.

Cement Behavior

Cement Shrinkage: If cement is placed at too high a water content, it loses water to the porous strata under lower pressure (p_o) through direct filtration because the cement hydrostatic head is greater than the pore water pressure head. The annulus width between casing and rock is small (e.g. 175 mm casing in a 225 mm hole = 25 mm), so even a small shear strength development between rock and cement will support the weight of the cement. If this shear stress is only -0.5 kPa, the entire "hydrostatic" head of the cement ($\gamma_c z$) can be supported by stress transfer to the rock mass. (Of course, because of temperature and pressure effects, this degree of cat is not attained simultaneously along the entire cement sheath.)

Thus, while the cement is still in an almost liquid, early-set state, massive shrinkage can occur by water expulsion, but annular cement settling to compensate for the loss of water is impeded by the shear stress transfer to the rock mass. The consequence is shrinkage in the cement sheath.

Portland cements continue to shrink after setting and during hardening.^{7,8} This autogenous shrinkage occurs because hydration reaction products occupy less volume than the original paste. Judicious proportioning control of the cement slurry and the use of admixtures and additives can limit the physico-chemical effects of the autogenous shrinkage processes. Mostly, the careful control of water content by using superplasticizers and the control of macro-shrinkage by using appropriate aggregates benefit the properties of the set grouts.

Silica flour (SiO_2 , ground to $-20 \mu\text{m}$) is often used to make "thermal cement". It is added in quantities approaching 75% of the dry constituents, the remainder being cement powder. Silica flour has also been added to cement in an attempt to counteract shrinkage. Unfortunately, for physico-chemical reasons, silica flour can enhance both drying and autogenous shrinkage.⁹

Silica flour is a ground product, usually made from pure quartz sand. Physically, the silica flour, by virtue of its grain size ($D_{50} = 10\text{--}20 \mu\text{m}$) has a large surface area; this provides

not only enhanced reaction areas for kinetically controlled hydration processes, it provides a need for additional wetting for slurry formulation. Physico-chemically, a freshly fractured silica surface possesses a high chemical reactivity because of the presence of unsatisfied bonds arising from the breaking of the silica chemical lattice. These fresh surfaces will electrostatically bind polar water molecules to satisfy these broken bonds. Experiments on pure silica using magnetic resonance and dielectric permittivity show that up to 9-11 layers of water can be absorbed on the surface, and the closest layers are of course the most tightly bound.

The surface area increases inversely as the square of the mean particle diameter, therefore reducing the surface area by a factor of five (grinding 100 μm sand to 20 μm flour) increases the area by 25, and because the new surface area is chemically fresh, it is more reactive. Thus, the electrostatic bound water volume for silica flour is vastly larger than for geochemically "old" sand. Furthermore, electrostatically bound water thickness is reduced by temperature (Brownian motion), so cool slurry will have a surfeit of water when it becomes heated through contact with geothermal temperature.

Alternative fillers are required to control the macro-shrinkage properties of the materials. We recommend 60-100 μm quartz sand be substituted for SiO_2 flour when possible.

Other processes can lead to cement shrinkage. High salt content formation brines and salt beds lead to osmotic dewatering of typical cement slurries during setting and hardening, resulting in substantial shrinkage.^{10,11} Experiments with recommended cement grout formulations placed against salt and potash strata clearly show massive dewatering of the cement and the formation of free brine at the interface between the cement and the salt. The same effect must occur when fresh-water cement grouts are in contact with low permeability rocks with highly saline pore fluids. By ensuring that the grouts are placed at high density, conducive to a stable grout microstructure, the effects of osmotic dewatering can likely be minimized, but this should be quantitatively assessed.

Recently marketed finely ground cements (Microfine™ and Ultrafine™) are Portland cement-based materials. They are generally finer than normal Portland cements and include pozzolanic additives, such as finely ground pumice. Slurries of these materials penetrate fine fissures and pores in rock more readily than more conventional grouts but in bulk suffer from very high shrinkage and, hence, without further modification, are not suitable for grouting the annulus between oil-well casings and the borehole wall.¹²

Dissolved gas, high curing temperatures, and early (flash) set may also lead to shrinkage. It is not clear if non-shrinkage additives have substantial positive effects at great depth and high temperature. These additives (e.g. Al powder) generally produce some gas, which in the laboratory provides volume increase. Additives may enhance some properties; however, they may induce negative impacts on other properties, or lose effectiveness at elevated temperatures, pressures, or in the presence of certain geochemical species. Also, autogenous shrinkage continues long after these agents have acted.

Cement Strength and Rigidity: API standards for oilwell cement specify certain strength criteria. Strength is not the major issue in oil well cementing under any circumstances. Based on extensive modelling, cement clearly cannot resist the shear that is the most common reason for oilwell distortion and rupture during active production.¹³ If compaction or heave (from solids injection) is taking place, the cement itself provides minimal resistance to buckling (compression) or thread popping (tension). If the annulus could be filled with relatively dense sand, the resistance to shear would be better than current ordinary oilwell cement formulations.

Based on over 50 triaxial tests at various confining stresses, we have shown that 28-day cured oilwell cements are contractile (volume reduction during shear) at all confining stresses above 1 MPa (150 psi). This is also the case for 70% silica flour cements, and for the new products based on extremely finely ground cement. (Specimens were cured under water at 20°C or at 90°C.) However, dense concretes used in Civil Engineering are dilatant, and therefore resistant to shear, at all working stresses.

The stiffness modulus of typical oilwell cement is small compared to that of low porosity rocks, and vastly lower than that of steel.¹⁴ The stiffness moduli are roughly 2-4% that of steel, though there is a wide range depending on density, content, and confining stress. Depending on depth (stress) and induration (porosity), rock moduli may vary from 2% to 50% of steel, and a reasonable value is 5-15% in most intermediate cases of moderate porosity (10-20%).

Bond. Cement will not bond to salt, oil sand, high porosity shale, and perhaps other materials. Also, bond strength (i.e. the tensile resistance of the cement-rock interface) is quite small; in fact, the tensile strength of carefully mixed and cured oilwell cement at recommended formulations is generally less than 1-2 MPa. Given that fluid pressures of 10's of MPa may have to be encountered, given that pressure cycling of a well can easily debond the rock and cement (there is strain incompatibility because of the different stiffnesses), and given that de-bonding is generally a fracturing process with a sharp leading edge rather than a conventional tensile pull-apart process, a large cement bond to rock cannot be assumed in any reasonable case. Initiation and growth of a circumferential fracture ("micro-annulus") at the casing-rock interface will not be substantially impeded by a cohesive strength at this interface.

The presence of "good bond" on a cement bond log is in fact not an indicator of bond, but an indicator of intergranular contact maintained by a sufficient radial effective stress. The lack of bond on a bond log is actually evidence of the inability to transmit high frequency sonic impulses because of the presence of an "open zone", that is, a circumferential fracture that is open by at least a few microns. Thus, maintaining "bond" actually means maintaining effective radial stress. Note that if effective radial stress cannot be maintained, then hydraulic fracturing conditions must exist at the interface.

The Gas Leakage Model

A good conceptual model must explain the following typical aspects of oilwell behavior that are observed in practice.

- Generally there are no open circumferential fractures detectable after a typical good quality cement job ("good bond" is observed on the log traces).
- Such fractures develop over time and with service.
- Even in cases where bond appears reasonable over substantial sections of the casing, gas leakage may be evidenced some years or decades later.
- The process is invariably delayed; thus, there must be physically reasonable rate-limiting processes.
- The gas often appears at surface rather than being pressure injected into another porous stratum encountered in the stratigraphic column.
- The presence of surface casing provides no assurance against gas leakage.

Whereas we do not deny that mud channeling, poor mud cake removal, gas channeling, and so on can occur in isolated cases, we believe that a better hypothesis exists to rationally explain the points listed above.

Figure 2 shows the effect of shrinkage on near-wellbore stresses. (Plots are qualitative, but have been confirmed by numerical modeling, to be published later.) Initially, cement pressure $p_c(z) = \gamma_c z$, almost always higher than p_o , but lower than σ_{hmin} (lateral minimum total stress). Set occurs and a small amount of shear stress develops between the rock and the cement; then, hydrostatic pressure in the cement is no longer transmitted along the annulus. Thereafter, even minor shrinkage ($-0.1-0.2\%$) will reduce the radial stress ($\sigma_r = \sigma_1$, p_o) between cement and rock because rock is stiff (4-20 GPa for softer rocks), and small radial strains (0.001-0.003) cause relaxation of σ_r and increase in σ_θ . A condition of $p_o > \sigma_r$ (σ_3) is reached; i.e. the hydraulic fracture criterion. A circumferential fracture (i.e. \perp to $\sigma_3 = \sigma_r$), typically no wider than 10-20 μ m, develops at the rock-cement interface.

A thin fracture aperture is sufficient to appear as "loss of bond" in a geophysical bond log. Because in situ stresses are always deviatoric (e.g. $\sigma_{hmin} \neq \sigma_{hmax}$), bond loss will usually appear first on one side of the trace, or on two opposite sides (direction of σ_{hmin}). Wells that have experienced several pressure or thermal cycles will almost always show loss of bond, sometimes for vertical distances in excess of 100 m.

A zone of $p_o > \sigma_r$ (σ_3) can extend for considerable heights. Nevertheless, this is still not a mechanism for vertical growth. To understand vertical growth, consider Figure 3, where a hypothetical case is presented. The static circumferential fracture of length L is filled with formation water of density γ_w , giving a gradient of about 10.5 kPa/m for typical oilfield brine, but the gradient of lateral stress ($\partial\sigma_r/\partial z$) is generally on the order of 18-24 kPa/m. This means that if the fracture contains a fluid pressure sufficient to just keep it open at the bottom, there is an excess pressure at the upper tip equal to $-L(21-10.5) =$ about 10 kPa/m, in typical Alberta conditions, for example. Thus, because of the imbalance between the pressure gradient in the fracture and the stress gradient in the

rock, an inherent fracture propagation force is generated that tends to drive the circumferential fracture upward. (In a perfectly horizontal section, this cannot happen, but the process develops equally at higher elevations in the well where it becomes inclined.)

Now, consider what happens when a circumferential fracture between the cement and the rock is exposed to a thin stratum that contains free gas (there are invariably several such zones in any well). Cementing a casing leads not only to the development of a cement sheath, but the cement paste also slightly penetrates the interstitial space in the surrounding rock (a few grain diameters deep for typical sandstone). This reduces the permeability substantially, and because of capillary exclusion effects associated with two-phase flow and the reduced pore throat diameter arising from cement particle invasion, gas flow into the circumferential fractures is almost certainly through diffusion. This means that when the fracture is small, the rate of gas influx is modest. However, as the fracture grows in height, the contact area with surrounding sediments increases, and eventually (and particularly when the pressures are being reduced by surface leakage or flow into a higher stratum), the gas diffusion rate is large enough to lead to continuous but slow gas leakage.

In the fracture, once solution gas saturation is achieved, free gas at the top of the fracture develops. The gradient in gas is less than 1 kPa/m (rather than ~10.5 kPa/m for water), so there is an even greater excess driving pressure at the upper tip. In addition, this gradient effect tends to favor driving the liquid in the fracture back into the formation, albeit slowly, and the fracture becomes more and more gas-filled. Thus, there is a self-reinforcing process; the greater the vertical height of the fracture, the greater the excess driving force at the tip. The fracture grows vertically upward, and eventually leads to gas leakage behind the casing at the surface. It will migrate up around the outside of any casing strings at higher elevations because the excess pressure that can be developed at that stage is large enough to fracture even excellent bond (Figure 4). However, why does it take so long for the gas to get to surface (sometimes decades)?

Gas must migrate to surface through a circumferential fracture perhaps only 10-20 μm thick extending over only a limited part of the circumference of the rock-cement interface. Note that fracture aperture develops between p_1 and σ_1 ($= \sigma_3$) when the pressure acts to maintain it open, but because the rock and cement have elastic stiffness, they act to severely restrict the aperture. Thus, there are at least two rate-limiting aspects to gas evolution at the surface: diffusion rate of gas into the fracture, and the low "hydraulic conductivity" of the circumferential fracture arising because of its narrow aperture.

Why does the fracture grow so slowly? When the microannular circumferential fractures are not connected and are short, the excess pressure at the tip is small. Also, if the casing pressure is large because of production pressure, this leads to a small outward flexure that may be enough to maintain the fissures closed. (Note that if a "better" bond log response is desired, simply pressurize the casing as the bond log is run!)

As the production pressure declines with time, the fissure will tend to open more because the casing is less pressurized. Also, fracture growth in the vertical direction is undoubtedly aided by pressure and thermal cycles.

Nevertheless, it is common for gas bubbling at the surface to be noticeable only years and sometimes decades after P&A. Over time, the effective fracture length increases, and this leads to the driving pressure increase discussed above. Because the velocity of a fracture is a very strongly non-linear process that is positively coupled to the driving pressure, it probably takes years for diffusion processes to lead to a condition where growth starts to accelerate. However, once acceleration begins, the fracture length increases, and complete upward propagation is fast (days? months?), limited only by the rate at which fluids can enter the fracture at depth and flow to the tip. Thus, before P&A, a cement bond log may show that the well is in good condition, yet this is no guarantee that, years later, leakage will not occur.

As the fracture rises, the condition that the pressure in the fracture exceeds the pore pressure in the surrounding strata will arise. This will lead to flow from the fracture out into the strata. If this flow is unimpeded, it will occur and the fracture vertical growth will terminate. Now, a condition exists where gas and liquids are entering the wellbore region behind the casing and leaving it at a higher elevation. This is a loss of zonal seal, and could have negative effects, such as pressurizing higher strata, or leakage of brines and formation fluids into shallower strata causing contamination. It can also have positive environmental effects, properly executed.

Yet, despite the existence of permeable zones, gas is still observed at the surface, and also as deep-sourced gas in shallow groundwater aquifers. The reason is probably that the cement paste in the pores of permeable strata acts to exclude gas by capillary effects along the entire length of the stratigraphic column (it takes a large Δp to overcome surface tension effects in small pores). This means that gas must leave the fracture mainly by liquid-phase diffusion. So, it seems that in leakage cases the flow rate from depth simply exceeds the diffusive bleed-off rate at higher elevations, leading to the excess appearing at the surface. An interesting chromatographic effect probably occurs with mixed gases; because of differing pressure solubility, more soluble gases will diffuse into adjacent strata more rapidly, and the least soluble, CH_4 , will arrive at the surface almost pure.

Unfortunately, even if no gas appears at the surface, it is no guarantee that the well is not leaking. In fact, the common occurrence of household water sources being charged with deep-sourced gas is clear evidence that there are many cases of leakage where the gas simply enters the water aquifer, and may never bubble around the casing.

Discussion

The hypothesis satisfactorily explains the phenomena associated with well behavior. Thus, it leads to a number of approaches to solve the problem. Eliminating cement shrinkage is one, but there are other practical solutions that are workable.

Cement shrinkage study and the development of new cement formulations that have no Portland phase¹⁵ is an ongoing part of an industry-sponsored project, and new formulations will be available soon. Better recommendations for P&A are also being developed. These will be the subjects of other articles. This section will present an approach to environmental protection that can be operationally implemented at present.

Given that gas leak-off by Darcy flow (rather than diffusion) is likely impeded by the cement paste in the pore space of adjacent strata, one approach to environmental protection is to complete a well in the manner sketched in Figure 5. The open, non-cemented section is deliberately chosen to be across beds of sufficient permeability so that when excess pressure develops in the zone, the capillary exclusion effect can be overcome (less than 1 MPa typically, but depending on grain size and clay content). Because the rate of gas entry and transmission through the circumferential fracture is small, a permeable bed just a few 10's of centimetres thick will suffice to act as a drain. This bed will accept sufficient volumes of gas, and providing that it is laterally continuous, will act as a drain for a very long time, perhaps indefinitely.

Is there a need to revisit API standards on cement formulation, placement and completion practices, and industry quality control during placement?^{16,17,18} We believe so, but this is a substantial issue, and specific suggestions await more results.

Closure

The elements of the gas leakage mechanisms that we propose are the following:

- Various mechanisms, but mainly cement shrinkage, lead to a drop in radial stress.
- When $\sigma_r < p_o$, a circumferential fracture will open.
- Differences between lateral stress gradients and pressure gradients provide forces for vertical growth.
- The excess pressure that develops at the upper leading tip increases as the (vertical) height.
- The fracture will tend to become gas filled as gas slowly diffuses into it, increasing the driving force.
- Fracture aperture is severely limited by the stiffness and geometry, limiting the upward propagation rate.
- Pore blockage because of cement paste penetration limits gas leak-off rates to those associated with diffusion because of capillarity effects.
- Eventually the fracture will rise, and gas will enter shallow strata or leak at the surface.

This working hypothesis has led to recommendations on cementing and casing strategies, and the pursuit of a cement formulation that can be easily placed yet not shrink is important, both for primary cementing, and for P&A.

References

- 1 Alberta Energy Utilities Board, 1997. Interim Directive 97-08. LONG TERM INACTIVE WELL PROGRAM REQUIREMENTS. Available from EUB, Calgary Alberta.
- 2 Ghofrani R. and Marx C. 1990. Special requirements for oilwell cements. In Properties of Fresh Concrete, ed. H. J. Wierig, Chapman and Hall, London, pp. 49-58.
- 3 Bonett A. and Pafitis D. 1996. Getting to the root of gas migration. Oilfield Review, Spring 1996, pp. 36-49.
- 4 Alberta Energy Utilities Board, 1999. Interim Directive 99-03. SURFACE CASING VENT FLOW/GAS MIGRATION (SCVF/GM) TESTING AND REPAIR REQUIREMENTS. Copy available from the EUB, Calgary Alberta.
- 5 Alberta Energy and Utilities Board, 1998. Application Number 970163, In the matter of an application by Imperial Oil Limited for an expansion... Available from AEUB, 10 volumes plus addenda, Calgary, AB.
- 6 Goodwin, K. J. 1997. Oilwell/gaswell cement-sheath evaluation. J. of Petroleum Tech., December, pp. 1339-1343.
- 7 Van Breugel 1991. Simulation of hydration and formation of structure in hardening cement-based materials - Delft, NL, 1991. ISBN 90-9004618-6.
- 8 Alcin, Neville, Acker 1997. Integrated view of shrinkage deformation - Concrete International, September 1997.
- 9 Gray, M.N. and Shenton, B.S. 1998. Design and Development of Low-Heat, High-Performance, Reactive Powder Concrete. International Symposium on High-Performance and Reactive Powder Concrete, Sherbrooke, PQ, Canada, 1998. August 16-20.
- 10 Van Kleeft, R. P. 1989. Optimized slurry design for salt zone cementations. SPE/IADC 18620. Drilling Conference, New Orleans, Louisiana, pp. 41-49.
- 11 Chenevert, M. E., Shrestha, B. K. 1991. Chemical shrinkage properties of oilfield cements. SPE Drilling Engineering, Vol. 6, No. 1, pp. 37-43.
- 12 Gray, M.N. and Shenton, B.S. 1997. Unpublished results of development tests on advanced finely ground cement grout mixtures. Report to the US/DOE, Sandia National Laboratories, Albuquerque, NM.
- 13 Dusseault, M.B., Bruno, M.S., Barrera, J., 1998. Casing shear: causes, cases, cures. SPE 48864. Proc. 1998 SPE International Conf. and Exhibition, Beijing, China, Nov 1998.
- 14 Thiercelin, M. J., B. Dargaud, J. F. Barret, and W. J. Rodriguez, 1997. Cement Design Based on Cement Mechanical Response. SPE 38598, Proc. Annual Technical Meeting, San Antonio, TX.
- 15 Bensted, J. 1996. Slag cement for oil well construction. World Cement, Vol. 27, No. 1, 6 pp.
- 16 API 1997a, Recommended Practice for Testing Well Cements. Recommended Practice 10B, 146 pages
- 17 API 1997b, Shrinkage and Expansion in Oil Well Cements, Technical report 10TR2, 57 pages
- 18 API 1999, Technical Report on Temperatures for API Cement Operating Thickening Time Tests. API 10TR3, 1st Edition, 100 pages

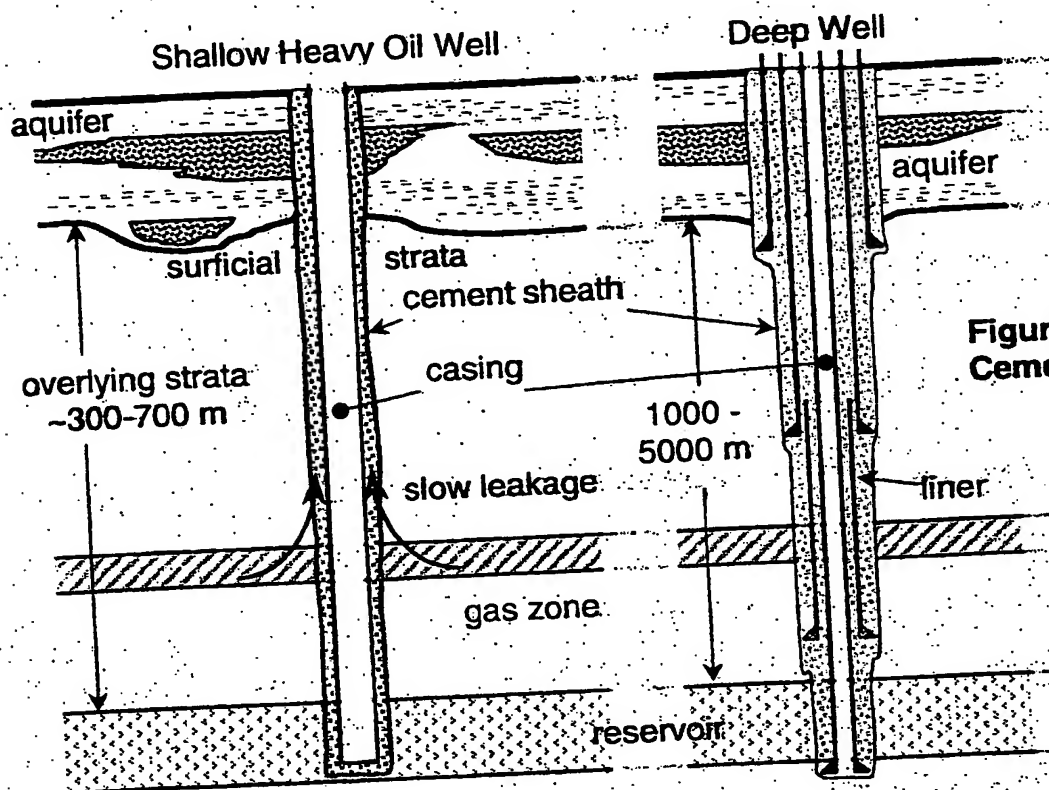
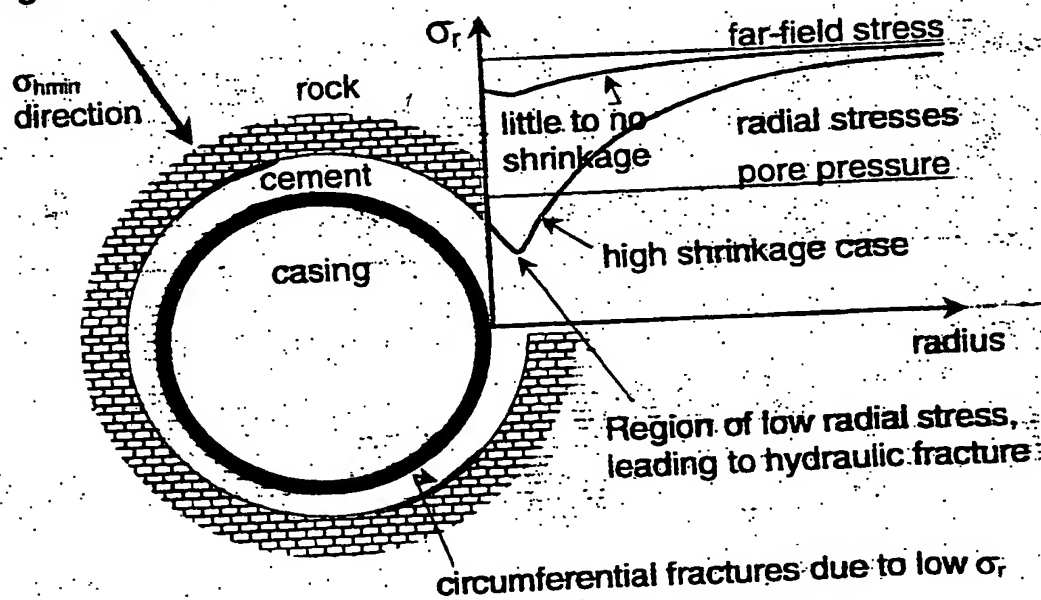


Figure 1: Cased, Cemented Wells

Figure 2: Radial Stresses and Circumferential Fractures



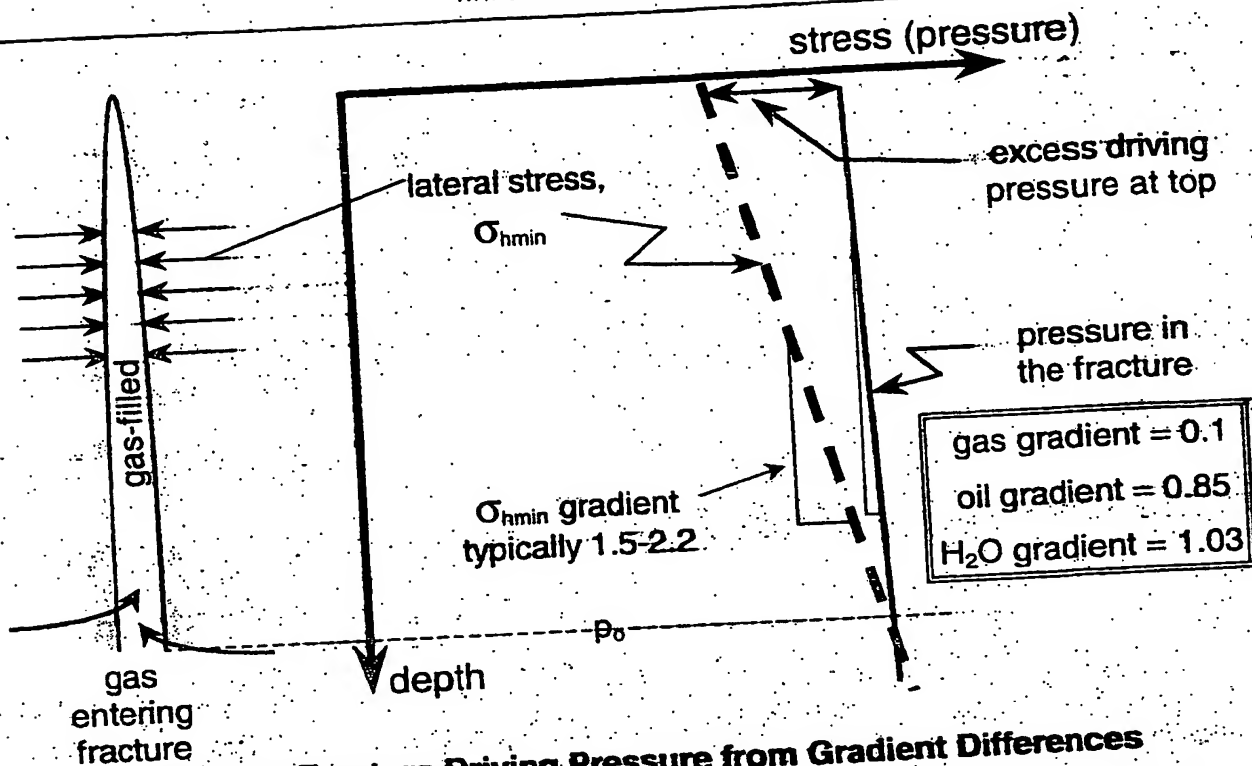


Figure 3: Fracture Driving Pressure from Gradient Differences

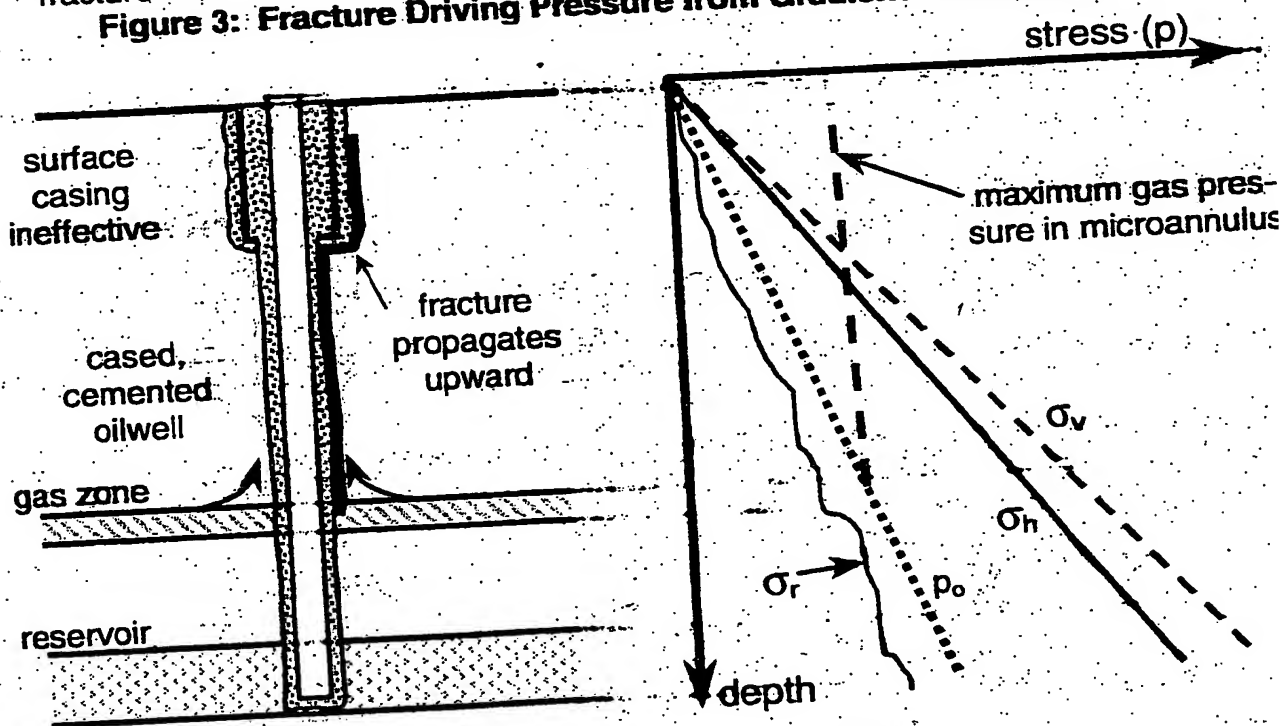


Figure 4: Fracture Approaching Surface

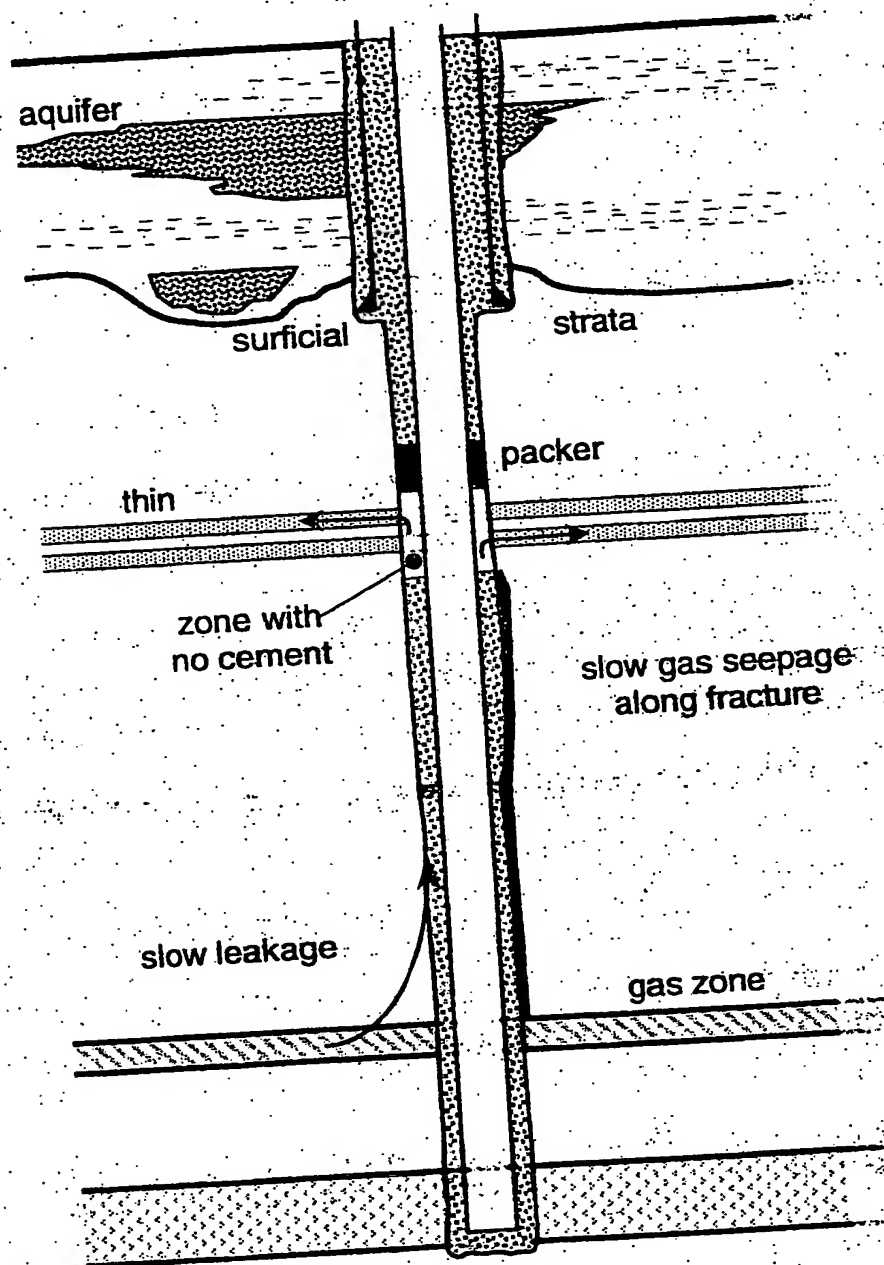


Figure 5: Leaving a Leak Off Zone to Arrest Gas Seepage

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US04/044048

International filing date: 29 December 2004 (29.12.2004)

Document type: Certified copy of priority document

Document details: Country/Office: US
Number: 60/533,824
Filing date: 31 December 2003 (31.12.2003)

Date of receipt at the International Bureau: 09 February 2005 (09.02.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

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